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SAPPHIRE MULTIPLE FILAMENT AND LARGE PLATE GROWTH PROCESSES

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Wright-Patterson Air Force Base
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SAPPHIRE MULTIPLE FILAMENT AND LARGE PLATE GROWTH PROCESSES

By

G. F. Hurley
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FOREWORD

This report was prepared by the Saphikon Division, Tyco Laboratories, Inc., Waltham, Massachusetts, under USAF Contract No. F33615-70-C-1471, Project No. 345-0, "Sapphire Multiple Filament and Large Plate Growth Processes." The work was accomplished under the technical direction of Lawrence Kopell, Project Engineer, Air Force Materials Laboratory, Manufacturing Technology Division, Wright-Patterson Air Force Base, Ohio 45433.

The authors – G. F. Hurley, S. Isserow, V. G. Kousky, H. E. LaBelle, Jr., and E. G. Roberge – wish to acknowledge the expert experimental assistance of H. Jeurs, W. Little, D. Meuse, J. Serafino, and V. White.

This report covers the results of the work carried out during the period 1 April 1970 to 31 March 1972. This report was released by the authors in August, 1972.

This technical report has been reviewed and is approved.

JAMES D. RAY, Branch Chief
Non Metals Branch
Manufacturing Technology Division
Air Force Materials Laboratory

ABSTRACT

Progress on concurrent programs to establish manufacturing programs for the growth of single crystal sapphire in two shapes is reported. One process is for the simultaneous growth of 25 high strength continuous sapphire filaments while the other is for the growth of 12 in. × 12 in. transparent sapphire plates. The objective of the first program was to increase the production rate capability and decrease the cost of sapphire filament by designing, building, and operating a multiple filament machine. The successful achievement of these objectives are described, and recommendations for scaleup to further increase production capability and to decrease costs are given. Particular advancements in the technology of several components used in the filament process are reported. The objectives of the second program were to develop a system and to establish a manufacturing process for the growth of large sapphire plates. The design and assembly of a vacuum enclosure and a new concept for heating the growth setup are described. The successful optimization of the equipment and procedures for the growth of intermediate size (5 in. × 10 in.) sapphire plates is discussed, together with the partial achievement of the complete objectives. Analysis of the remaining optimization for the growth of high quality, full-scale plates is reported.

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I. INTRODUCTION

A. Background

The initial impetus for the development of single crystal sapphire filaments was based on its potential as a structural reinforcement. Single crystal sapphire has high modulus and high strength, which are retained to high temperatures. The chemical properties are also attractive, since sapphire is highly stable and has much less tendency than other reinforcing materials to undergo degradative interactions with matrix materials.

In a series of programs, Tyco Laboratories, Inc., has advanced the technology of growing continuous lengths of small-diameter, single crystal sapphire filaments. Initially, under an AFML-sponsored feasibility study [AF No. 33(615)-3237], Tyco produced 6-in. lengths of 5- to 20-mil filament from the melt. Further studies under a follow-on contract (AF No. F33615-67-C-1098) led to two significant improvements. First, the "self-filling tube"* feeds liquid to a growth interface that is maintained at a fixed location in a constant environment. Second, the cross section of the growing crystal is kept constant by the "edge-defined, film-fed growth" (EFG) technique. This technique is not limited to fine filaments. In fact, as seen below, it has been successfully applied to prepare significant lengths in other configurations. Commercial production of continuous filaments was brought closer to reality in a program sponsored by the Manufacturing Technology Division (AF No. F33615-68-C-1126). A belt pulling mechanism was developed to withdraw continuous lengths of the growing crystal. It was also shown that, in a batch operation, seven filaments can be grown simultaneously from a linear array of orifices in the same melt. One of the objectives of the present program is to reduce the cost of sapphire filaments by extending the technology to the establishment of a continous multifilament process.

As noted in the preceding paragraph, the EFG technique is applicable to configurations other than filaments. Articles already prepared include ribbons, tubes, and plates for applications such as microelectronic substrates, lamp housings, and armor plate. An example of

*U.S. Patent No. 3,471,266, October 7, 1969.

particular interest is a sapphire plate 3 in. × 3 in. × 0.25 in. which, when polished, transmits light very effectively (AF No. 33615-69-C-1369). Larger plates are of potential use for transparent armor. This program therefore had the second objective to extend the technology to large plates and to establish a manufacturing process for such plates.

B. Program Scope

The present program was performed to establish manufacturing processes for two shapes that can have considerable use: (1) filaments, and (2) plates. The filaments were to have diameters of 5 or 10 mil. The plates were to be 12 in. × 12 in. with a thickness of 3/8 in. and 3/4 in.

Since the essential features of continuous filament production had been demonstrated, it was necessary to effect such production in a manner that would substantially reduce the cost of the filament and thus permit its broadened application. Cost reduction was achieved by two types of economies: (1) improvements in operations for individual filaments, and (2) the simultaneous processing of a large array of filaments. In contrast, the process for large plates is a scale-up of techniques developed for small plates.

For growth of the filaments and the plates, separate tasks were set up for assembly of appropriate machines. In Phase I of each task, the design features were established. The various devices were designed and constructed, and the performance of each such device was evaluated. In Phase II of each task, the machines for each type of growth were set up and operated. Alternate approaches which had been considered during Phase I were used during the more extensive testing of Phase II to optimize growth conditions. Both machines were operated nearly continuously for several weeks at the end of Phase II to demonstrate their reliability. In addition, the filament machine was used for a production run during which more than 20,000 ft of sapphire filament was produced. The mechanical properties of this filament were determined by tensile testing.

C. Organization of Program and Reports

In Phases I and II, each major task (filaments and plates) consisted of a set of subtasks corresponding to the processing steps or major items of equipment. In this report, each major task is discussed separately, with each section subdivided into the major items of equipment and processing steps.

II. SAPPHIRE FILAMENT PROCESS

A. Introduction to Section II

This section of the report covers the sapphire multiple filament program and discusses the various accomplishments and problems encountered during the program. Primarily it evaluates the multiple filament growth process and the multiple filament machine system. It also discusses the results of the production run, quality control procedures, system operating procedures, and filament characterization. Finally it summarizes the results of the program and discusses the requirements involved in the scale-up of the process to a larger quantity of filaments.

B. Program Objective

The general objective of this program was to design, develop, and construct a continuous multiple filament growth machine with the capability of continuously growing, pulling, coating, and spooling 25 single crystal sapphire filaments. These filaments were to be 0.010 ± 0.0005 in. diameter and to have average strengths of 400 KSI at 25°C. In addition, these filaments were to have a density of 3.98 g/cc and an elastic modulus of 65×10^6 psi at room temperature. These filaments were to meet the specified requirements for strength, elasticity, and density, with a minimum yield of 80%. The finished machine was to have a production capacity of 170 lb/yr of 10 mil diameter filament. The ultimate goal of the program was to reduce the cost of filament by a factor of 400 from the basic price of $75, 000/lb for laboratory produced single filament.

The program has been successful in meeting the basic objectives. The basic purpose of the program objective was to make possible reduction in the price of the filament. Indeed, an order of magnitude reduction in the price of filament was implemented before the conclusion of this program based on technical improvements resulting from the early work on the program. At the present time, the objective of reducing the per pound cost to the vicinity of $200 can be considered to be achieved under the condition that enough of a market is generated to warrant operation of 3 or more 25 filament machines.

Some problem areas still exist, but it is felt that with further development these could be corrected or at least minimized to the point where the effect on production would be negligible. These areas are discussed in detail in this report.

C. System and System Evaluation

This section describes the multiple filament machine (Figs. 1a and 1b) and evaluates the performance of the various components of the system. The system is based on simultaneous growth from 25 filament orifices in a single crucible. Although components of the system are accordingly based on the apparatus for the growth of a single filament, a number of these components were designed in such a manner that significant improvements in function were achieved with concommitant improvement in economics and quality of the filament. In particular, the belt puller, the setup design incorporating much lower cost insert orifices, and the rigid guide all represent large improvement over their respective antecedents.

In general the system is capable of growing, coating, and spooling 25 filaments simultaneously. Due to certain inadequacies the system is not completely automatic (although this was not a basic requirement of this program) but requires monitoring by an operator to make power level and growth speed adjustments to maintain ideal growth conditions. With further development effort, however, completely automatic operation could be attained. Also, with hindsight, it is apparent that certain features could have been improved if a different design approach had been taken, mostly in the area of ease of operation.

The following is a description and evaluation of the individual components of the system.

1. Filament machine frame

The main objectives in the design of the frame (Fig. 2) were to provide a rigid, stable structure upon which the system components could be mounted such that the various operating functions could be performed easily and quickly by the operator. In this design, rigidity and stability are attained by using structural members of sufficient size and mass. The frame consists mainly of two basic assemblies: (1) the lower base platform, which includes two thick steel plates and four solid steel legs, and (2) the upper superstructure, which is secured to the base platform by bolts. Two 7 ft long, 1-1/2 in. diameter steel shafts are used to maintain alignment between the various system components directly related to the growth operation. The entire unit is mounted on four vibration-isolating mounts which damp out vibrations from the floor. The frame was designed to have such a large mass (approximate weight 3000 lb with all components mounted) that any accidental jarring or external excitations would have no effect on the growth operation. Further to nullify vibrations, the pulling mechanism platform, the guide positioning platform, and the crucible/susceptor pedestal platform are mounted to the parallel shafts by means of special isolation mounts. Included in the design of the frame is the stereomicroscope positioning mechanism which allows the operator to switch very quickly from one viewport to the other to check filament-to-orifice alignment during the critical seeding operation.

Also, the frame has been designed so that all of the various components for positioning the guide, operating the puller, and operating the coater are within easy reach of the operator. The crucible/susceptor assembly is mounted on a platform that slides up and down on the

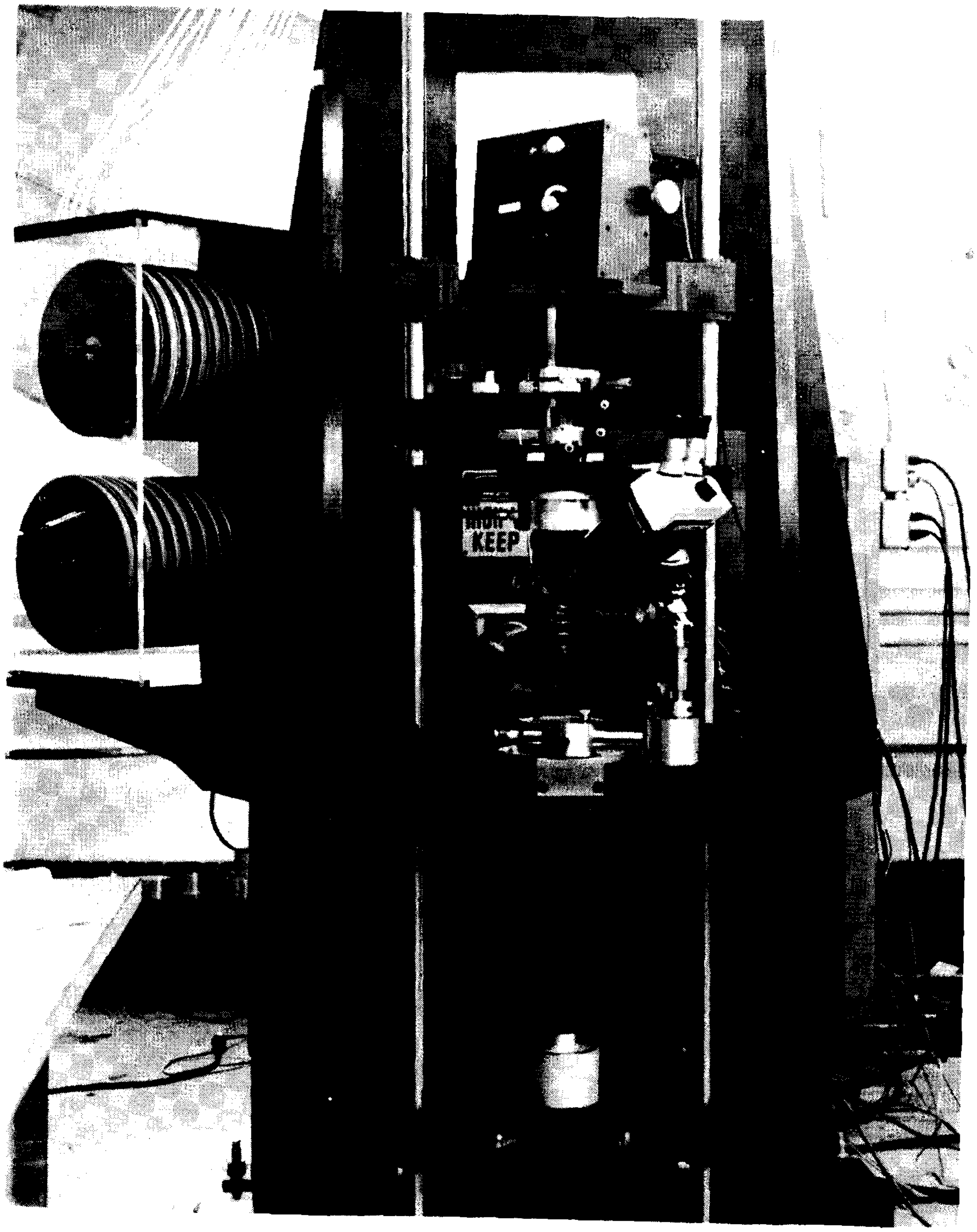


Fig. 1A. Sapphire filament machine assembly

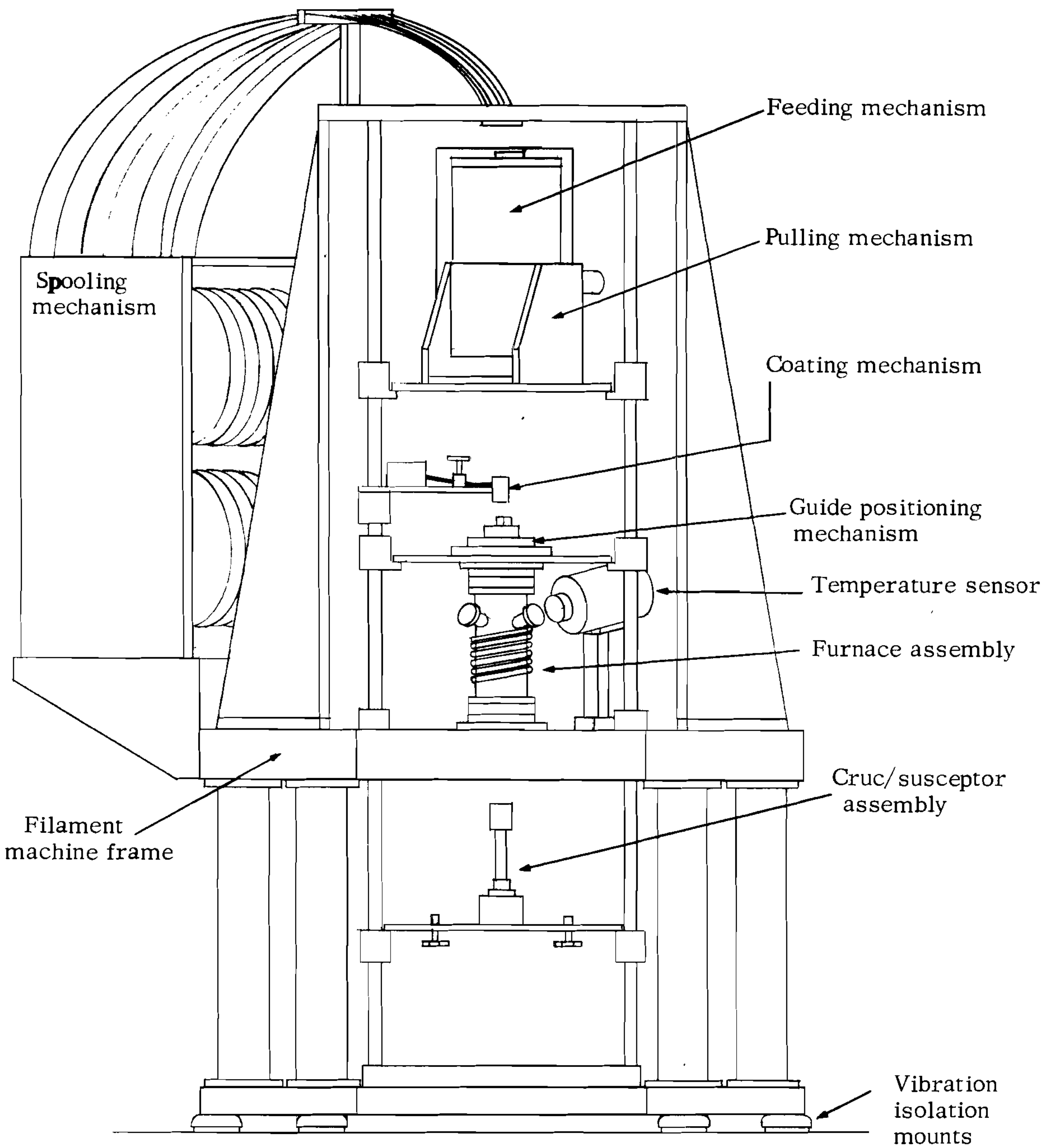


Fig. 1B. Sapphire filament machine assembly

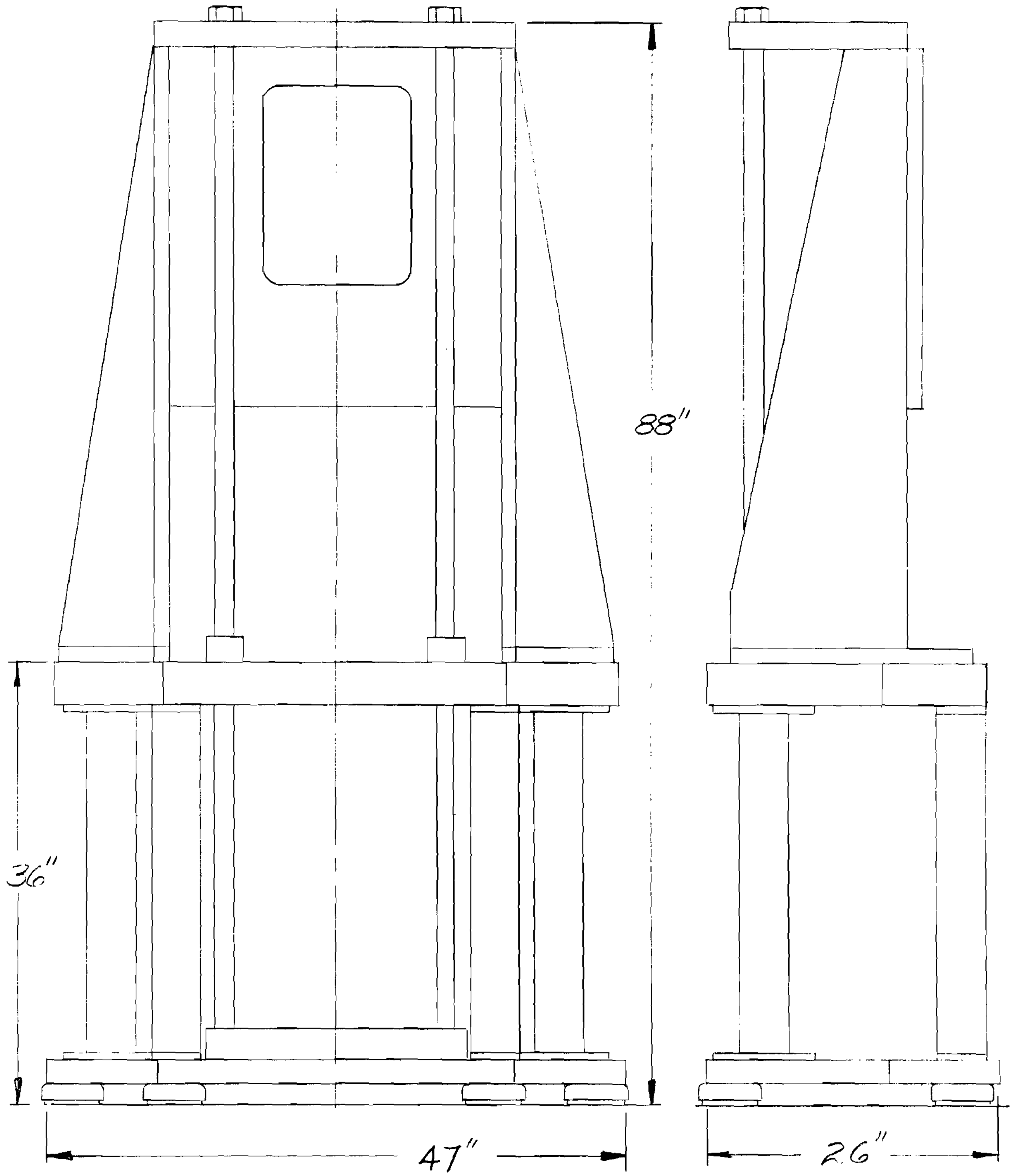


Fig. 2. Filament machine frame

parallel shafts, allowing for easy removal and installation. This platform is counterbalanced by two spring reel assemblies so that its movements are controlled by a slight hand pressure. In its operating position, this platform is clamped to the main base by two large bolts and consequently it is extremely stable and free of any perturbations.

Another feature of this design is that any of the various components can easily and quickly be removed for servicing without affecting the alignment of the other components. For example, the quartz furnace chamber can be taken out of the system simply by removing six bolts and sliding it out; it is not necessary to remove the guide positioning mechanism.

The frame can be considered successful in having met its performance requirements, specifically in the areas of providing vibration isolation and alignment of the critical components.

2. Heating power unit

The present method of producing sapphire filaments is through the use of induction heating. It was determined on a previous contract (AF Contract No. F33615-68-C-1126) that resistance heating, using a tungsten mesh element, was inferior. Adjustment of the temperature distribution was difficult, and maintenance and reliability proved to be major problem areas. Induction heating, on the other hand, is being used successfully in the growth of sapphire plates, ribbons, and tubes. On this basis, it was decided at the outset to proceed with induction heating. The correctness of this decision was established early in the program and has been confirmed by all subsequent experience.

To keep the power requirements at a minimum, it was decided that using a graphite susceptor and graphite insulation provided the most efficient arrangement for our application. We have proceeded on this basis, and sufficient test data have been obtained to verify the validity of our basic assumptions. The 450 kc Lepel generator that we are using is rated at 20 KW (output power) and is more than adequate for our needs.

The induction heating unit (rf generator) for the multiple filament operation is a Lepel Model T-20-3-KC-RP-5W manufactured by Lepel High Frequency Laboratories, Woodside, New York. This unit takes the low frequency (60 cycles) alternating current from the power line and passes it through an electronic tube generator which converts it to a high frequency signal. This high frequency current is then conducted through leads to the load coil.

The induction heating unit specifications are as follows:

Power input: 440 V-3 phase-60 cycles (approximately 44 KVA)

Power output: 20 KW at 450 kilocycles

Controls:

Power output: Stepless thyratron control – 0 to maximum output
Main power supply: Magnetic trip circuit breaker
Filament power: Pushbutton switch

Controls (continued)

Plate power: Pushbutton switch

Grid current control

Tubes: Two oscillators/three rectifiers/three thyratrons

Water requirements: Oscillator tubes – 4 gals/min
Tank circuit and load coil 1-1/2 gals/min
Pressure – 35 to 60 psi
Temperature – not to exceed 86°F.

Some difficulties were encountered in the operation of the Lepel rf generator unit in the form of blowing fuses, tripping out the safety relay, etc., due to internal arcing and shorting out. This can be attributed to several factors, notably the age of the unit, inadequate maintenance of the unit, and a hostile environment due to the presence of graphite fibers and particles in the atmosphere. These particles and fibers were being drawn into the generator by the suction of the internal cooling fan and, as they settled, they provided electrical paths for the high voltage present, thereby causing arcing. After the system was thoroughly cleaned, marginal components replaced, and filters installed to block the entrance of particles, the unit performed satisfactorily.

In a regular production operation, especially if a larger quantity of filaments was being grown, the reliability of the power unit would be essential to the operation. Therefore, a regular maintenance program is required and should include a periodic cleaning and overhaul of the unit.

The present unit, rated at 20 KW output power, operates at less than 50% of its capacity. Therefore, this size unit is capable of providing sufficient power for producing a much larger quantity of filaments. The exact number would be determined by the design of the orifice array and the resultant crucible size, the type of shielding and insulation, and the size of the furnace chamber.

3. Furnace chamber

The furnace chamber (Fig. 3) is a double-walled Pyrex glass enclosure which has water flowing between the inner and outer walls to remove heat radiated by the setup. The water enters the base through the water cooled fittings, flows upward between the walls, and exits through the upper fitting. Circumferential O-rings in the fittings are used to seal the waterpath as well as provide a nonrigid support for the chamber. The unit has an 80-mm I. D. and a 100-mm O. D. Three 28-mm I. D. viewports with sealed quartz windows provide adequate viewing for seeding and observing growth and also provide sighting access for the optical pyrometer. When installed, the chamber provides an airtight enclosure with the only access to the atmosphere being through the guide slots. Here the flow of argon into the chamber provides very slight gauge pressure and prevents air leaks through the guide slots.

The design of the furnace chamber has proven to be satisfactory, and any further scaleup would employ the same basic design. The present size can accommodate a 2-in. O. D. crucible, while a 125-mm O. D. chamber could accommodate a 3-in. O. D. crucible.

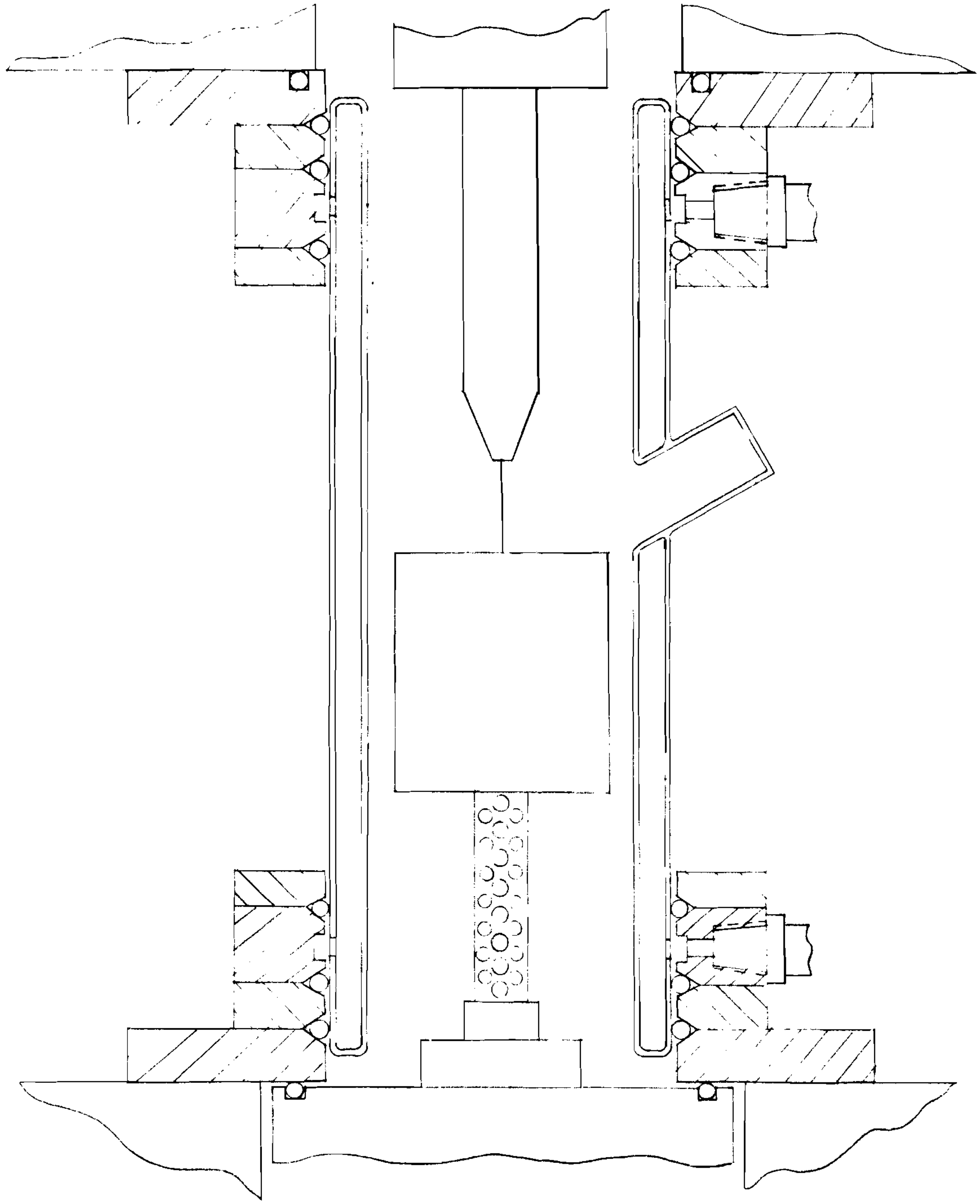


Fig. 3. Furnace chamber

4. Temperature control system

The temperature control unit for the multiple filament growth system maintains the operating temperature of the crucible at a constant level (i.e., within prescribed control limits) by sensing the temperature of the crucible. According to the temperature sensed, the control unit supplies input commands to the rf generator to increase or decrease the power level to correct for any deviations from the preset temperature setting. An optical pyrometer, Ircon Model 1240, is used to sense the crucible temperature and a controller unit, Electromax Model 6261, is used to regulate the rf generator.

The Ircon optical pyrometer consists of a sensing head and an indicating unit joined by an interconnecting cable. The sensing head contains focusing optics and a detector in a cast aluminum housing which compensates for ambient temperature over the range 20°F to 160°F. The sensing head measures the infrared energy emitted by the setup by focusing its objective lens onto the detector. Focusing is accomplished by the operator sighting through the eyepiece in the back of the unit and adjusting it until the target of interest (e.g., the crucible lid) is brought into focus in the reticle. An integral optical filter limits the spectral content of the radiation being measured. The detector then generates a current proportional to the radiation impinging on it. This signal is transmitted to the indicator unit via the interconnecting cable.

The indicator unit receives the signal from the sensing head and, through a precision feedback operational amplifier, increases the signal level to a usable magnitude. The signal is then divided into an output to a temperature-indicating meter and a signal to the control unit. The indicator unit operates on 115 Vac and contains an internal power supply to drive the various components. Operating specifications are as follows:

Calibration accuracy: 1% of full scale temperature

Repeatability: 0.5% of full scale temperature

Sensitivity: 0.2% of range

Response time: at outputs, 0.5 sec (95% of final value)

Output: 0 to 5 Vdc (to controller)

The temperature control unit (Leeds & Northrup Model 6261 Electromax C. A. T. controller) is a solid state instrument that provides current-adjusting-type (C. A. T.) control to adjust its dc output current to the level required for maintaining the input equal to a set point. The input signal comes directly from the Ircon indicator unit and the set point is determined by the operator. This setting is accomplished through a multiturn potentiometer and indicated by a four-digit counter on the front panel. Any deviation of the input emf from the set point emf is indicated by a zero-center deviation meter on the front panel. This input signal emf is opposed by the set point emf that represents the temperature at which the crucible is to be controlled. Output current is then applied to the rf generator circuit, thereby regulating the output power to the induction coil.

This system has performed with some degree of success but has not proven adequate for completely automatic temperature control over long periods of growth. Mainly, the problem is in the proper sensing of the operating temperature at the growth interface. Sighting at a remote location such as the radiation shield has proven not to be satisfactory for the degree of control required for this operation using the particular design setup involved. The temperature must be monitored directly at the growth area. This poses a problem for two reasons: first, the viewing area of the optical pyrometer is too large to focus directly on a single ideal spot such as the meniscus of one filament or a particular surface on a single orifice; second, during the growth process sapphire droplets are deposited over most of the visible surfaces, changing the emissivity of the surface being measured and therefore the apparent temperature reading. During the production run it was found that better results were obtained using manual control, with the operator making the necessary power adjustments by observing the filament diameters and making the corrections to maintain the proper growth temperature. Also, it became apparent that the setup became more temperature sensitive with the advent of the final design insert orifice which has a smaller body diameter, and therefore less mass, than the previous design (which proved unsatisfactory for other reasons). Indications are that it is possible to reduce the temperature sensitivity at the growth interface by applying some minor design changes to the setup. One specific example would be to increase the body diameter of the insert orifice slightly, which could be accomplished without any major change in the basic setup design.

Completely automatic temperature control was not achieved on this program. However, sufficient experience has been obtained with the system to say, with a reasonable degree of confidence, that with further more extensive development, automatic temperature control could be achieved to the point where the unit could be left unattended for long durations.

5. Filament guide

The filament guide is a 12-in. long piece of high density graphite 1.25 in. × 0.75 in. in cross section. The guide is made in two parts which are held together with tungsten pins. One half contains 25 precision machined slots, 0.0135 in. × 0.0135 in. and spaced 0.040 in. apart. The The other half has a flat surface so that when the two halves are joined together they form the guide slots for the filaments. The guide is secured in a water-cooled guide holder in the guide positioning mechanism and extends from outside the furnace chamber down into the chamber to a point 1.75 in. above the orifice array. The function of the guide is to position the filaments in the proper vertical alignment with regard to the orifice array and, during growth, to provide the guidance necessary to maintain the filaments in line with the respective growth interfaces. Due to the action of the argon passing up the slots as it escapes from the furnace chamber, the slots can be considered similar to gas bearings, where a cushion of gas is present between the walls of the slot and the filaments. The slot width for the growth of 0.010 in. filament was 0.0135 × 0.0135 in., and was derived through experimentation.

At the beginning of the contract, an additional objective was to grow 0.005 in. diameter filament. Accordingly, another guide to have been used here was purchased and had 0.007 ×

0.007 in. slots, also spaced at 0.040 in. apart. It was felt that this size would provide sufficient clearance for accommodating minor misalignment and still provide guidance as in the case of 0.010 in. filament. The 0.005 in. filament guide was never used, however, as this objective was deleted during the contract period.

The rigid guide has been proven successful in this program. It provides adequate control of the filament during growth and, with proper seeding, no problems are experienced in transporting the new growth junctions through the guide. Graphite has proven to be a satisfactory material for the guide. It has good thermal characteristics, does not damage the filament, and has reasonably good wear characteristics. The particular graphite used in our applications is Airco Speer Carbon Products Grade 9326.

In any scale-up of the multiple filament process, a rigid guide using the same design principles would be used.

6. Guide-positioning mechanism

The guide-positioning mechanism (Fig. 4) is located directly above the furnace chamber and is mounted on the 1-1/2 in. diameter parallel shafts of the filament machine through specially designed vibration-isolating mounts. The top water-cooled fitting flange is bolted to the guide positioning mechanism, and a floating O-ring seal is used to maintain the airtight integrity of the furnace chamber. This unit is a precision machined mechanism which holds the guide in vertical alignment while providing fine adjustment capability in the horizontal plane. It has two mechanical stages to provide lateral movement in the X and Y directions and a third component to provide rotational adjustment. Precision-ground ways are used to insure accuracy in lateral movement, and two precision radial bearings are used to maintain the vertical accuracy of the rotational component. In addition, the unit contains a water jacket which cools the guide during operation and thereby maintains the temperature of the mechanism at a constant level. This is necessary because otherwise the heat transferred from the furnace would heat the mechanism, make it too hot to handle or function properly, and would degrade the O-ring seal. Micrometer heads are used in conjunction with strong compression return springs to provide the necessary fine adjustment.

The performance of the guide-positioning mechanism has been satisfactory in that it meets all of its basic design requirements. In retrospect, however, it would appear that a fixed alignment arrangement would be a better method for alignment of the filaments to their respective orifices. At the time that this component was developed, it was not practical to use a fixed alignment arrangement. The problem with the present arrangement is that only a skilled operator can accurately position the filaments in proper alignment with respect to the orifice array, and this operation remains basically a matter of judgment. The problem is compounded when the filament seeds do not meet the straightness requirements. Certainly, in any scaleup of the multiple filament operation a fixed alignment method would be required whereby the guide and orifice array would be pre-aligned so that no adjustments would be necessary. This could be accomplished by supporting the entire crucible/susceptor assembly from the guide using a transition piece. Tungsten locating pins could be used to align the orifice array with the guide

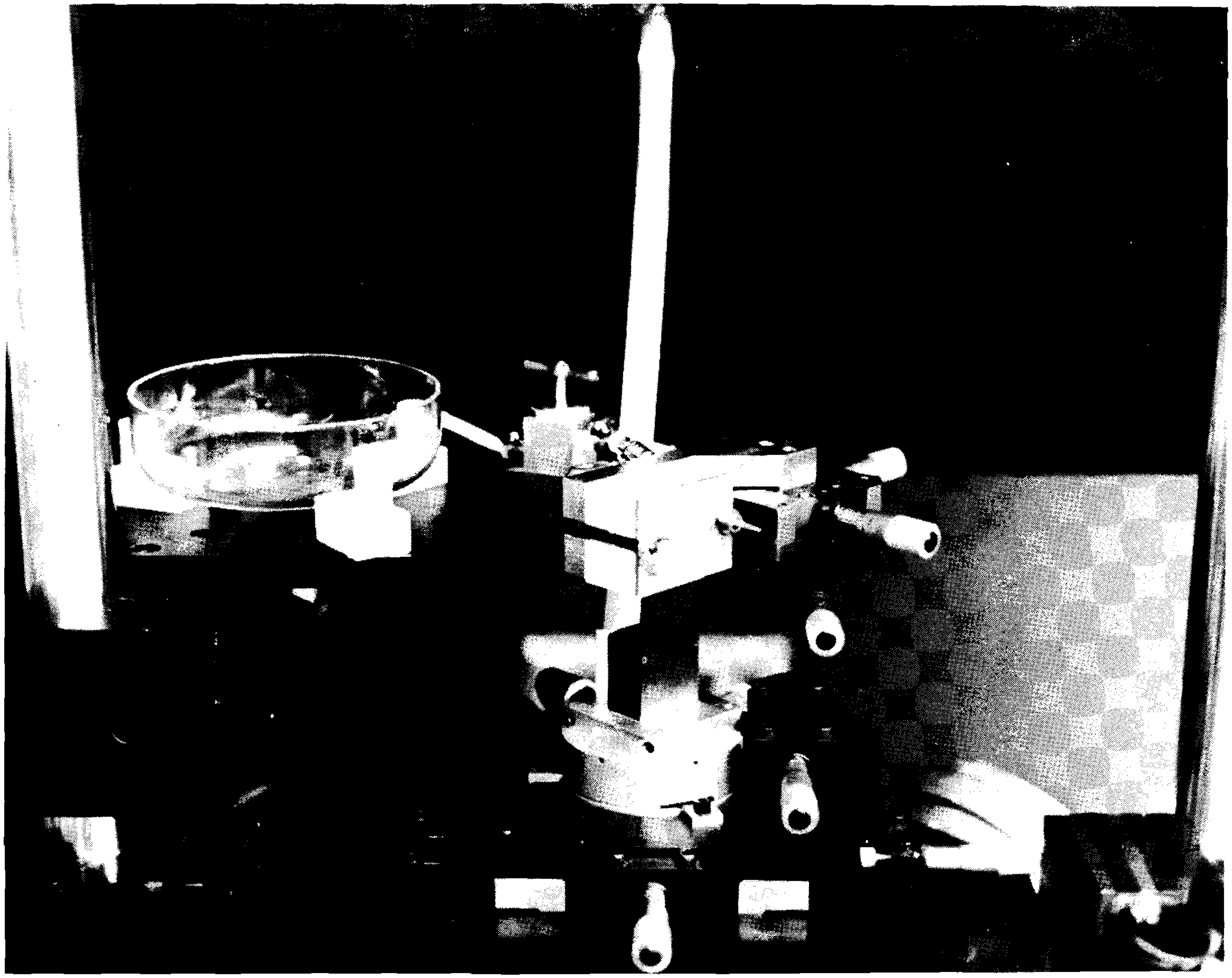


Fig. 4. Guide positioning and coating mechanism

slot array. Design and development efforts would be required to compensate for the effects of expansion of the various components and also to make the necessary modifications to correct any thermal imbalance caused by the additional components in the induction coil susception zone.

Aside from the aforementioned aspects of the fixed alignment setup, the modified alignment feature would permit another simplification of the machine. In addition to the fact that no guide positioning mechanism would be required, the lower carriage assembly for the setup pedestal could also be eliminated. The guide/setup assembly could be loaded into the top of the furnace chamber as one unit.

7. Continuous feed mechanism

The continuous feed mechanism (Fig. 5) for the multiple filament operation functions automatically to replenish the supply of Al_2O_3 in the crucible as it is drawn out in the form of growing filaments. The feeder unit consists of a rotating disk with pockets machined in its periphery which pick up sapphire granules from a feed chamber and drop them into a feed canal to the crucible. The feed rate is determined by the rotating speed and the number and the size of the pockets. This unit has a 1/2-in. thick disk with 12 pockets. The pockets are machined such that the granules are retained as the pocket is moving upward, yet they are able to slide out (and into the exit port) as the pocket is moving downward. This wheel is mounted on a shaft which is supported by two radial ball bearings in the feed chamber wall. A worm gearset couples the wheel shaft to the drive motor. A reservoir chamber, which is an integral part of the feed chamber and extends above the wheel, replenishes the sapphire supply as it is removed. The wheel is "immersed" in the granules so that in the pickup area the level of the sapphire is above the midpoint of the wheel.

The drive system for the feeder consists of a variable speed dc motor and a speed control unit. The motor is mounted on the baseplate outside the feed chamber and is coupled to the worm gearset through a vacuum-tight feedthrough. The motor is rated at 1/50 horsepower. The worm gearset reduces the speed at the wheel to 1/5 rpm and increases the torque at the wheel. The speed control unit is mounted on the system control cabinet front panel and has an off/on control and speed control adjustment which has been calibrated to establish the various feed rate settings. The feeder assembly is mounted on a baseplate and is enclosed in a glass bell jar. The baseplate contains feedthroughs for the vacuum and gas lines. In operation the unit is sealed and the air evacuated using a vacuum pump. The latter is carried out to remove the water vapor adsorbed on the sapphire. After evacuation, the bell jar is filled with argon at a slight positive pressure.

The feeder is mounted on the filament machine above and behind the furnace chamber. The feed path to the crucible consists of a 1/2-in. I. D. Tygon tube which connects the feeder outlet port to a 1/4-in. O. D. sapphire tube extending from outside the furnace chamber to a point directly above a feedport in the crucible lid.

The performance of the continuous feeding mechanism has been satisfactory. There has been no adverse effect on the growth process due to the feeding of crushed sapphire granules to the melt during operation of the system. The only change that would be recommended would be to relocate the feed chamber to a more accessible position of the frame. The present design

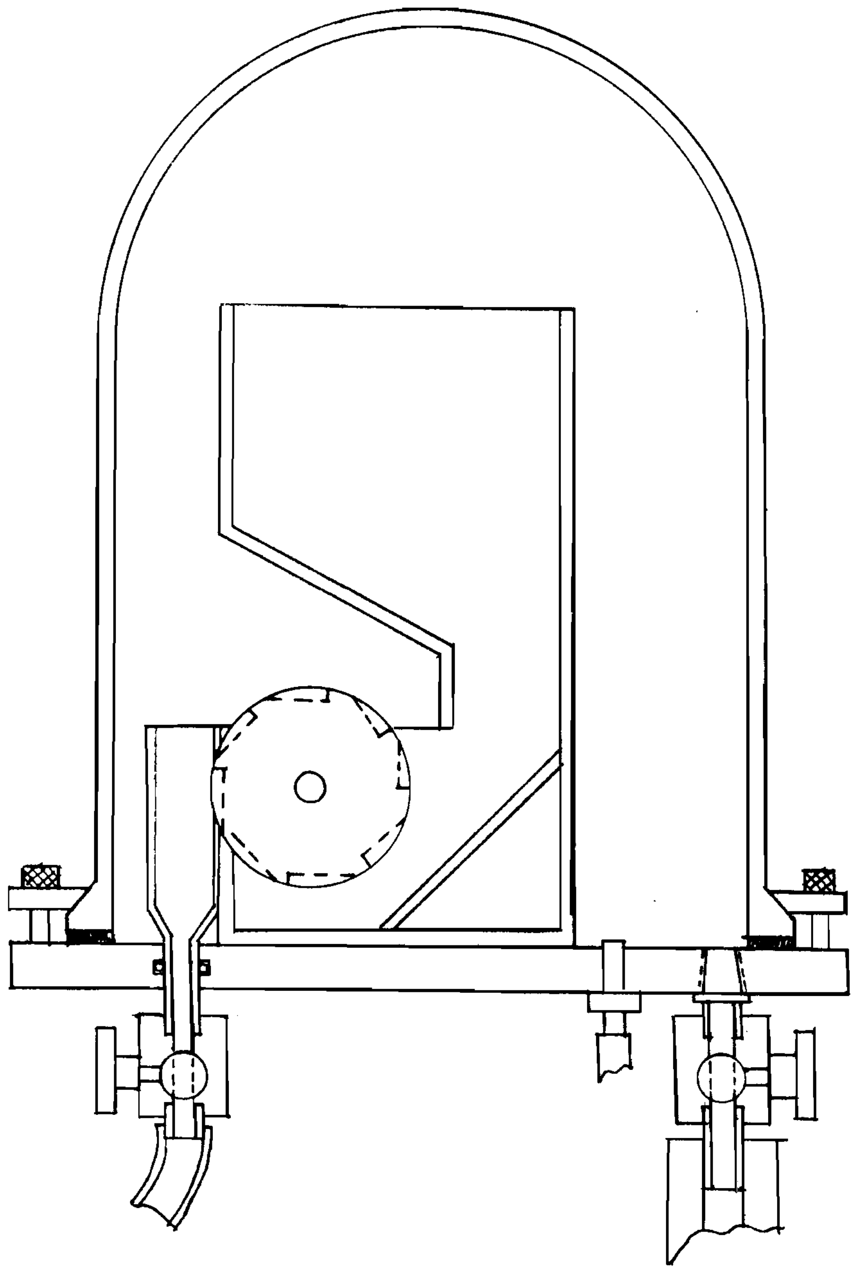


Fig. 5. Continuous feeding mechanism

unit is adaptable to a scale-up system as it now stands. It has the capability of providing melt replenishment for a substantially higher rate-volume.

8. Pulling mechanism

The function of the pulling mechanism is to grip the filaments and pull them straight upward from the growth interface at a steady rate, with the pulling action being smooth and free of any cogging effects, jerking, or perturbations of any kind. This unit must travel in reverse as well as forward for the seeding and growth operations, respectively, and the pulling speed in both directions must be variable to allow the operator the required flexibility to establish the seeding and start of growth functions.

The pulling mechanism for the 25-filament operation (Fig. 6) is a belt-type unit with the belts being friction driven on their respective rollers by a dc velocity servo through a gear train. The belts are of the endless closed loop type and travel in counter-rotating directions so that in the area where they contact the filaments they are traveling in the same linear direction (see Fig. 7). Each belt contacts and travels over four separate rollers: a drive roller, a pressure roller, a tension roller, and a idler roller. The drive rollers are connected to the gear train and provide the input power which transports the belts. To insure that the belts will not slip, pressure rollers are used to press the belts against their respective drive rollers. The pressure rollers are spring loaded to obtain the pre-set pressure required. The tension rollers exert an outward force on their respective belts so that proper belt tension is maintained and the belts do not have any slack. The function of the idler rollers is to position the belts in the proper operating location. These rollers are located in line with the drive rollers so that the proper spacing between the belts is achieved. This spacing is 0.060 in. when the belts are in their open position, i.e., when no pressure is applied to them by the backing plates. All the rollers except the pressure rollers are machined with shoulders on each end so that the belts are nested between the shoulders and consequently are restrained from moving off their rollers. The rollers are mounted on precision ground shafts which fit into precision-tolerance radial ball bearings. The bearings are pressed into the front and back plates of the pulling mechanism, which have been line bored to insure perfect alignment of the rollers with respect to each other. This is necessary to obtain proper belt tracking.

The belts themselves are a silicone/glass fabric manufactured by Dodge Industries, Hoosick Falls, New York. This material is an extremely fine weave fiberglass cloth which is impregnated with silicone rubber. Its properties include high yield strength, a non-stick surface, and resistance to wear and abrasion. The assembled belt is 1-1/4 in. wide × 17 in. long and 0.020 in. thick. It is made by bonding together (with a silicone adhesive) two 0.010-in. thick strips of Dodge M301 silicone/glass fabric terminating in an angled butt joint to maintain a uniform belt thickness.

Backing plates (see Fig. 8) are used to apply pressure to the back side of the belts so that the belts will grip the filaments and transport them in the desired direction. The plates have a Teflon facing in the area of belt contact so that the sliding friction between the plates and the belts is minimized. Each backing plate is supported by four precision-ground shafts which travel on precision linear ball bearings. A compression spring on each shaft is used to

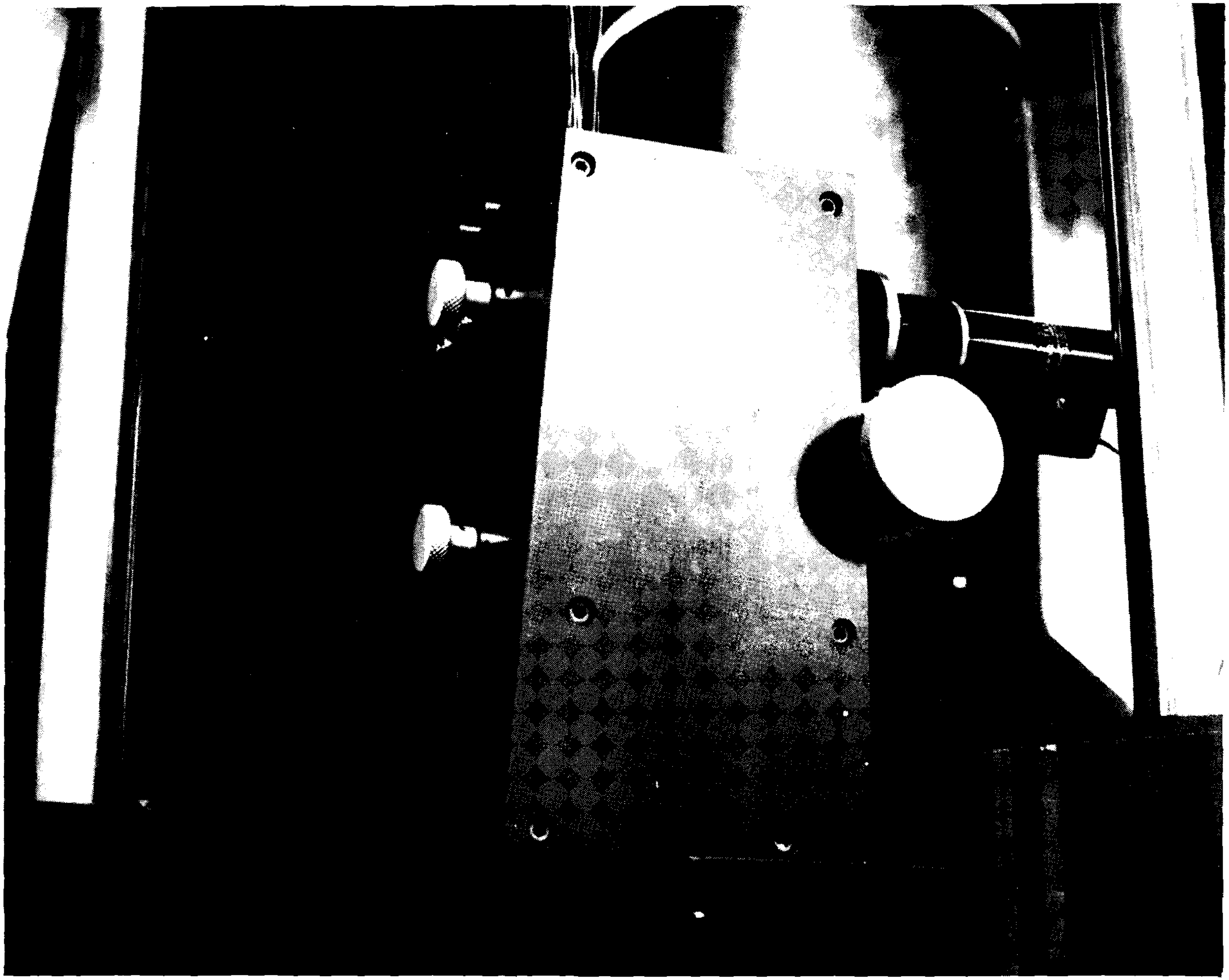


Fig. 6. Pulling mechanism

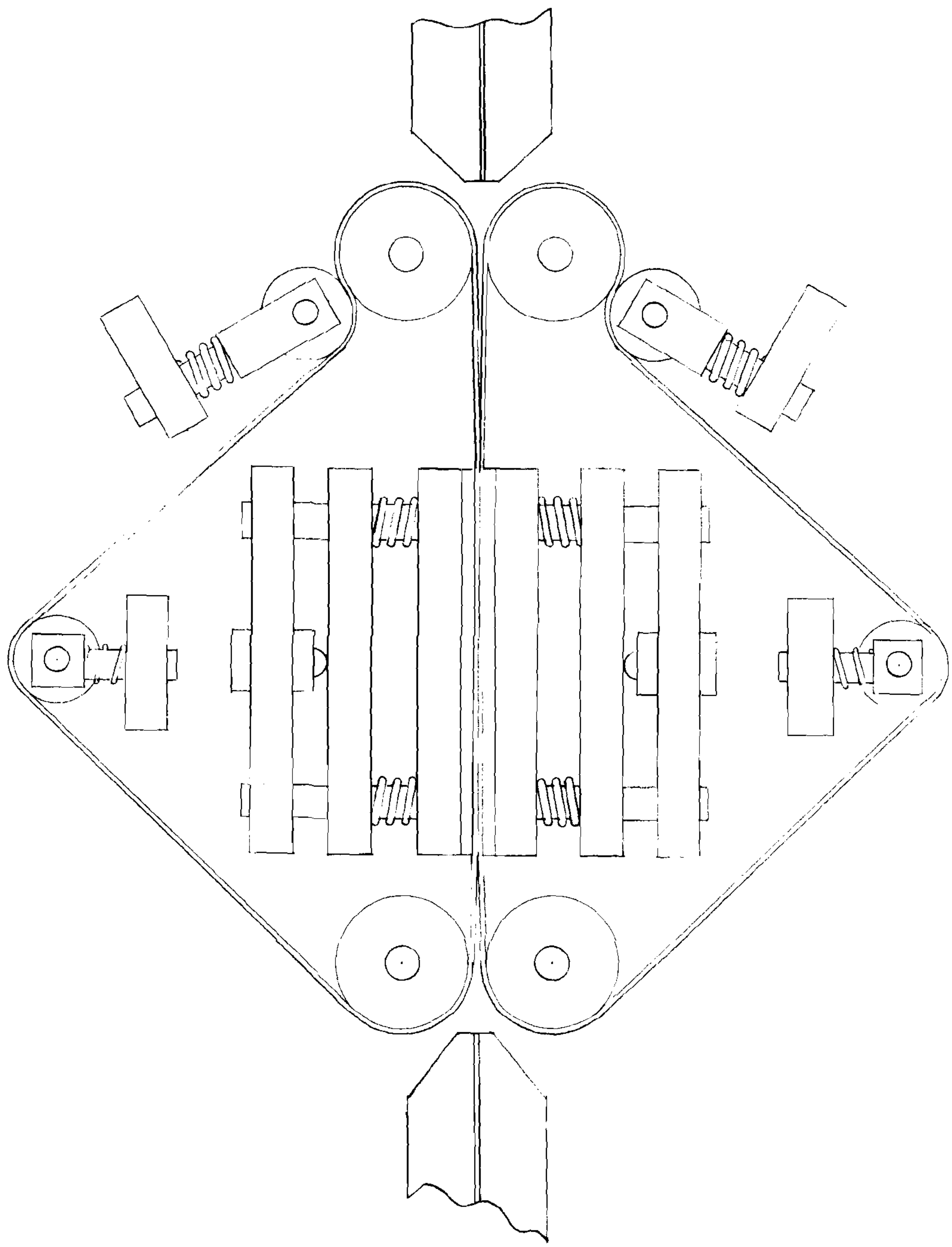


Fig. 7. Pulling mechanism – belt drive assembly

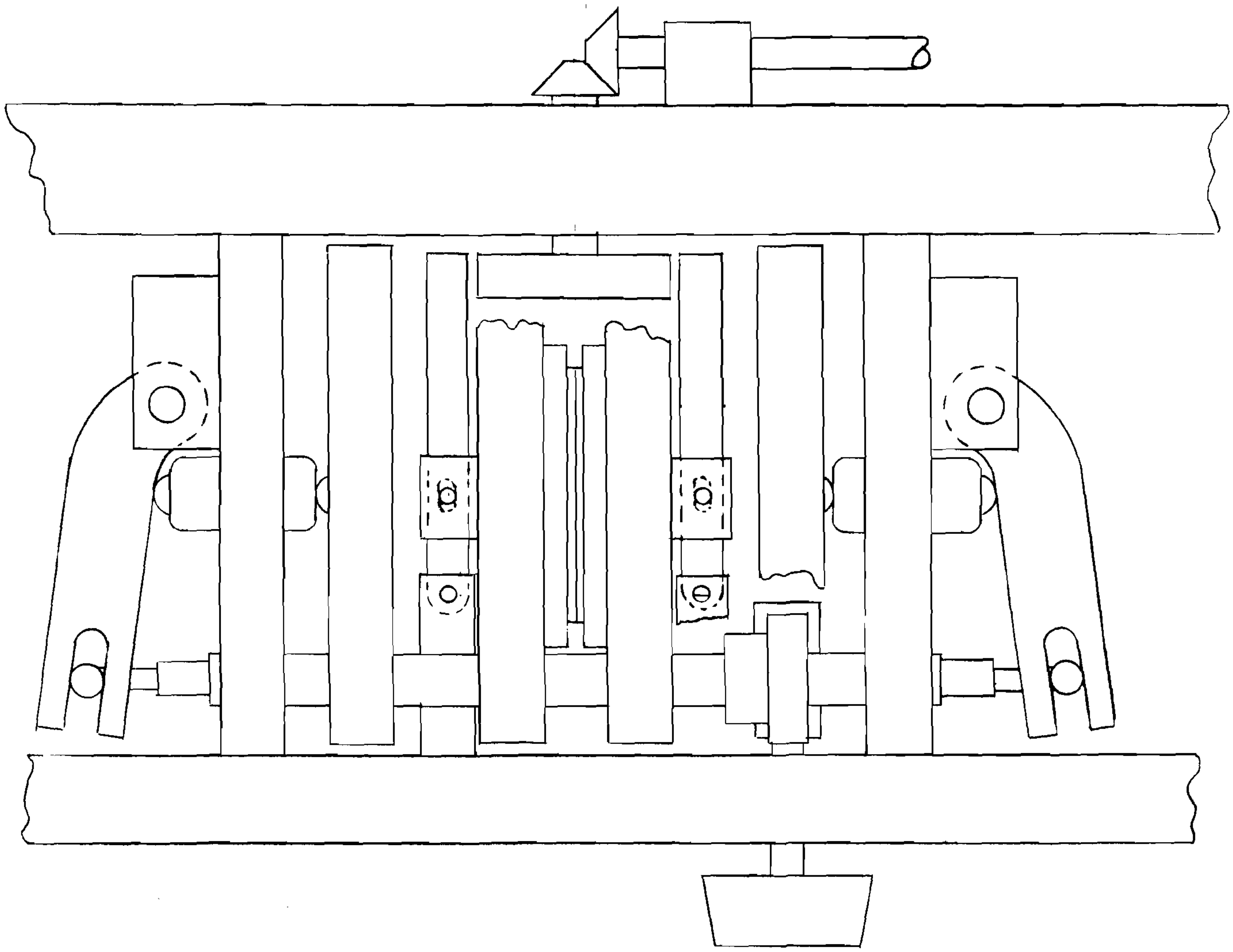


Fig. 8. Pulling mechanism – backing plate assembly

apply a constant pressure to the backing plate. Uniform compression of each spring is obtained by use of a sliding plate which is positioned by a plunger sliding on a bearing in the mounting plate. The position of the plunger is regulated by a mechanical linkage which, in turn, is positioned by a clevis pin riding in a slot in the linkage. The lateral position of the clevis pin is controlled by the movement of the threaded rod which, in turn, is controlled by rotating the threaded bushing. This is accomplished by the operator, who turns the knob connected to the worm gear. Since each backing plate has its own individual pressure adjustment controlled by the same source, the same pressure loading is applied to both plates simultaneously. This arrangement provides an extremely fine pressure adjustment, with over 80 turns of the knob required to go from zero pressure to full pressure. Also, if desired, the pressure range can be changed or shifted simply by changing the compression springs. Equally important is that this design maintains the backing plates perfectly parallel to the belts so that even pressure is applied over the entire contact area.

A quick-release device is included in this mechanism and moves the backing plates away from the belts to allow the filaments to be inserted or removed. This is accomplished with a cam/linkage arrangement which, when actuated, moves the backing plates away from the belts. A quarter turn of the quick-release knob is required to release the belts. When the knob is turned clockwise this motion is transmitted to the cam via a shaft and a set of bevel gears. The cam then acts on the linkage which, in turn, through clevis pins riding in slots in the linkage arm, moves the backing plates away from the belts by compressing the springs. An advantage of this design is that the pressure setting is not changed, and when the quick release is deactivated, the backing plates return to their original setting.

The drive system for the pulling mechanism is composed of the dc velocity servo and a helical gear train. The dc velocity servo consists of a torque unit and a control unit. The torque unit contains a permanent magnet dc motor, a dc feedback generator, and a speed-reducing gearhead. Armature speed is reduced by a factor of 200:1 to provide a maximum output speed of 18 rpm. The control unit contains a zener reference voltage supply, a transistor control network, a power supply, a power amplifier, a potentiometer, a control switch, and a speed-indicating meter. The unit operates in the following manner. The switch is set in the operating mode (either forward or reverse) and the potentiometer is set to achieve the desired speed setting. The power supply, consisting of a transformer and a full-wave rectifier supplies dc power to the dc amplifier which in turn supplies power to the motor in accordance with an input signal. To make the motor rotate, a command signal is obtained from the combination of the zener reference voltage and the variable potentiometer that is set to the desired speed setting. To achieve high accuracy the servo loop is closed by using a speed signal from the temperature-compensated, directly driven dc tachometer generator. This output voltage is connected so that it opposes the potentiometer command voltage. The differential or "error" voltage (i.e., the difference between the potentiometer command signal and the tachometer feedback signal) is then impressed across the input of the dc amplifier. Since the amplifier requires only a change of a few microamperes to vary the input power to the motor, this arrangement ensures that the motor has the precise power required to maintain its speed at a selected value regardless of the torque loading. The same tachometer feedback signal is also used to motivate the speed-indicating voltmeter directly to indicate motor speed.

The torque unit is mounted on the back plate of the pulling mechanism. Power is transmitted to the drive rollers through a helical gear train. This gear train provides a further reduction in speed of 4: 1. Helical gears are used to reduce backlash to a negligible amount. The speed range at the drive rollers is infinitely variable from zero to 4.5 rpm. A slip clutch is used to connect the main drive gear to the drive shaft so that the unit can be driven by hand. A knob on the front plate is connected to a spur gear which meshes with another spur gear on the drive roller shaft. By turning the knob the belts can be hand driven, without damaging the motor gearhead, since the clutch will slip.

The pulling mechanism is 9-1/2 in. high × 10-1/4 in. wide × 5 in. deep. The housing is constructed from 3/8-in. aluminum plates. Graphite filament retainers are used directly above and below the belts to maintain proper spacing between the filaments as they pass between the belts.

The pulling mechanism is mounted on the filament machine directly above the guidance mechanism. It is mounted on a platform which, in turn, is mounted to the two 1-1/2 in. diameter shafts by means of special vibration-isolation mounts. These mounts are secured to precision linear bearings so that the entire platform can be moved up and down freely. Specially designed shaft locks are used to secure the platform at the desired location on the shafts. The pulling mechanism is mounted on the platform at a 45° angle (to the centerline of the machine) so that the plane of the belts will coincide with the plane of the filament array as it leaves the guide.

The pulling mechanism has been successful in meeting its design requirements for performing the pulling function of the multiple filament growth operation. Successful multiple filament growth could not have been accomplished without this unit. It provides a smooth pulling action (due to the friction drive design and the servo controlled torque unit) and firm gripping of the filament array so that the filaments do not slip in the belts. The design of the puller is adaptable to being scaled up to pull a larger number of filaments simply by increasing the size of the belts and the various backing plate components. In the event of a scale-up of the multiple filament system, serious consideration would be given to the use of belt pullers of this basic design to perform the pulling action. The design of the pulling mechanism for a scaled-up system would be influenced by many factors such as the number of filaments to be grown, the orifice array pattern, how many groups of filaments to be pulled by one drive system, etc.

9. Crucible/susceptor assembly

The crucible/susceptor assembly, shown in Fig. 9, consists of a graphite support pedestal, graphite susceptor, crucible assembly, radiation shields and graphite felt insulation. The assembly is mounted on a movable platform so that it can be lowered for

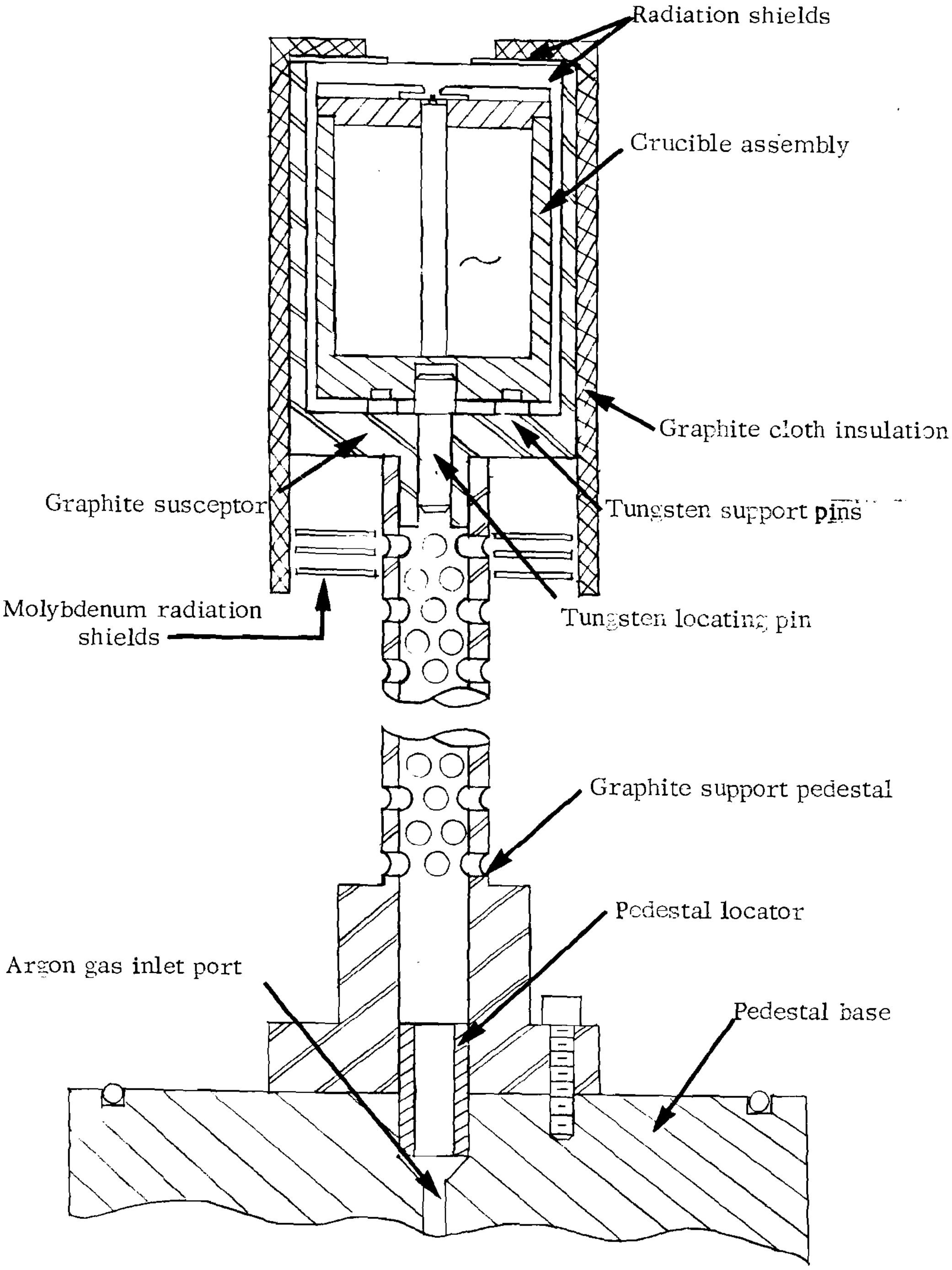


Fig. 9. Crucible/susceptor support assembly

removal from the furnace chamber. The crucible/susceptor assembly is bolted to a mounting base on the platform which, when in the operating position, is sealed at the bottom of the furnace chamber by means of an O-ring butting up against the bottom of the furnace chamber lower fitting. Argon is introduced into the furnace chamber through a feedport in the pedestal base and flows upward into the support pedestal and out into the chamber through the holes in the tubular portion of the pedestal.

The support pedestal is machined from a single piece of high purity graphite to precision tolerances. It is secured to the base by three cap screws and is located concentric to the growth axis by a locating pin in the pedestal base. The susceptor is mounted into the support pedestal by a slight push fit into the top of the support pedestal. The crucible is supported in the susceptor by three tungsten pins and is located concentric to the growth axis by a tungsten locating pin. The primary radiation shield (molybdenum) is mounted directly on the crucible lid and is located relative to the orifice array by four pins which are pressed into the lid. The secondary tungsten radiation shield is placed on the top of the susceptor. Graphite felt insulation is used to cover the top, bottom, and sides of the susceptor. Three tungsten radiation shields are mounted on the support pedestal directly below the susceptor to reflect the heat being radiated from the bottom of the susceptor.

The crucible assembly (Fig. 10) consists of the crucible lid, orifice/capillary mount and the primary radiation shield. It is commonly referred to as the "setup." The crucible is machined from solid molybdenum rod stock and is 1.750-in. O. D. × 1.938-in. overall height with a 1/8-in. wall and an inside depth of 1.625-in. The crucible lid is 3/16-in. thick and is recessed 1/16-in. deep into the crucible by means of a machined shoulder which locates the lid concentric to the centerline of the crucible. With this thickness lid no distortion has been experienced with a thinner lid design. The orifice/capillary mount assembly is positioned in the setup so that its feet rest on the bottom of the crucible and it is held in proper vertical alignment by the crucible lid. The orifice/capillary mount assembly is secured in position by machining the slot in the lid so that a slight push fit is required for installation. The height is set such that the top of the capillary mount is 0.005 in. below the top surface of the lid.

The primary radiation shield is made from 0.060-in. thick molybdenum and consists of two semicircular sections. In the area of the orifice array the bottom of the shield halves have a machined recess 1.85 in. wide × 1.50 in. long × 0.20 in. deep. When installed in position, the shield halves form a 0.040-in. groove in which the orifice array is located. The edges of the shield adjacent to the orifice array have a 30° × 0.015 in. bevel so that proper viewing of the orifice tips can be achieved. The bevel is preferred to the use of a thinner lid, since this thickness (0.060 in.) is required to prevent distortion during operation.

In addition, the shield is machined so that the primary radiation surface to the orifice array tapers in height from 0.025 in. in the middle of the shield to 0.020 in. at the outer edges. This taper serves the function of making heat loss in the vicinity of the orifices a function of radial distance from the center orifice. This pattern of heat loss is required by the direction

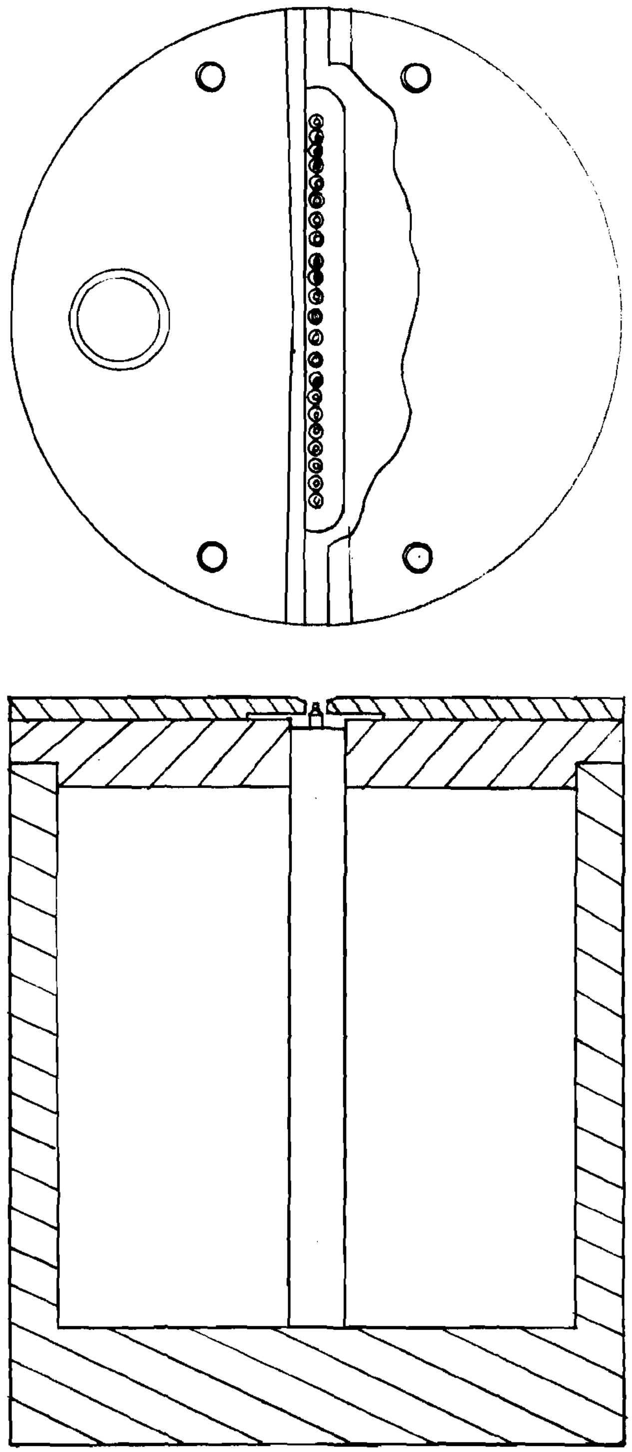


Fig. 10. Crucible assembly

of heat flow in the setup. Heat flow is from the edges of the crucible toward the center and, with no compensation, would follow a temperature gradient increasing along the line of orifices toward the edge of the crucible. By allowing heat loss above the crucible lid (which is carrying heat toward the center), and by making these losses greater near the extremes of the line of orifices, the temperature gradient along the orifice tips can be made level. This design gives filaments of equal diameters at all the orifices.

The orifice/capillary mount assembly (Fig. 11) consists of the 25 insert orifices contained in a linear array by a mount which also provides the capillary path for the molten sapphire to flow to the orifice tips. The spacing between the individual orifices at operating temperature is 0.040 in. and coincides with the spacing of the guide slots. To achieve proper positioning at operating temperature, the orifices are spaced 0.0393 in. apart at room temperature to compensate for thermal expansion. The capillary mount consists of two 0.060-in. thick molybdenum plates, 1.250 in. wide × 1.750 in. high, and two 0.025-in. thick spacers. The plates have a 0.040-in. × 0.008-in. deep groove running across its width and 0.015 in. down from and parallel to the top of the plate. When the mount is assembled with the spacers, these grooves form a channel into which the insert orifices are loaded in their proper configuration. The correct spacing is achieved by maintaining the major diameters of the orifices at 0.0393 ± 0.003 in. and securely butting them together during installation. This design also maintains the orifices in proper vertical alignment. Loading of the orifices is quite easily accomplished and the method used does not damage the tips. The mount is assembled using three lightly-pressed rivets to hold the assembly together. This allows one of the spacers to swing out of the way so that the orifices can be inserted into the grooves. The individual insert orifices (Fig. 12) are installed using tweezers. During installation they are given a final check for height uniformity. Then the spacer is swung back into its final position, the orifices are squeezed together so that they are all butted together, and the assembly is riveted together.

The insert orifice shown in Fig. 12 is the final design orifice used to grow 0.010 in. filament. The tip dimension is critical here as the filament is grown with a diameter strictly less than the tip diameter. This design allows freedom to grow filament in the range up to approximately less than 0.020 in. by changing the tip diameter. A set of insert orifices blanks (orifices are received without the tip hole which is drilled here) was ordered in the early part of the program for the growth of 0.005 in. filament, but were never used.

The design of the crucible/susceptor assembly evolved over the duration of the program. Many design changes were initiated to correct problem areas, such as the distortion of various components and inadequate alignment of the orifice array, and also to achieve the thermal equilibrium required in this assembly. The cost factor also had a large influence in the evolution of this design. For the production of filaments to be economically feasible, it was necessary to design this setup so that production costs would not be prohibitive. The present design can be considered successful in meeting the functional aspects as well as the cost aspects of the design requirements. The necessary alignment tolerances of the orifice array have been

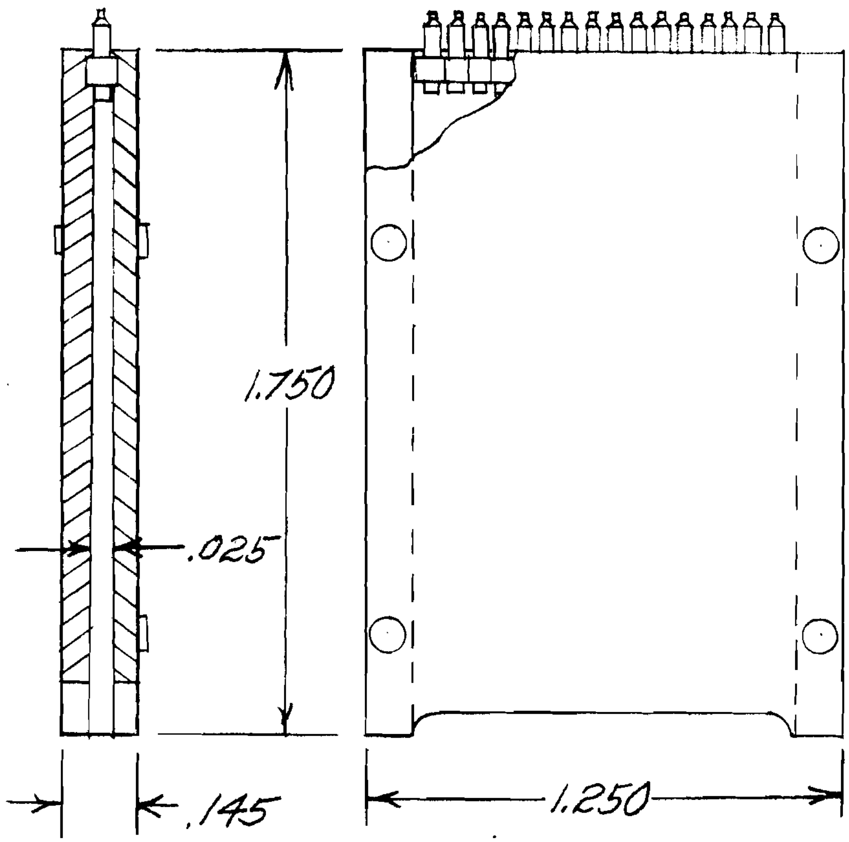


Fig. 11. Orifice/capillary mount assembly

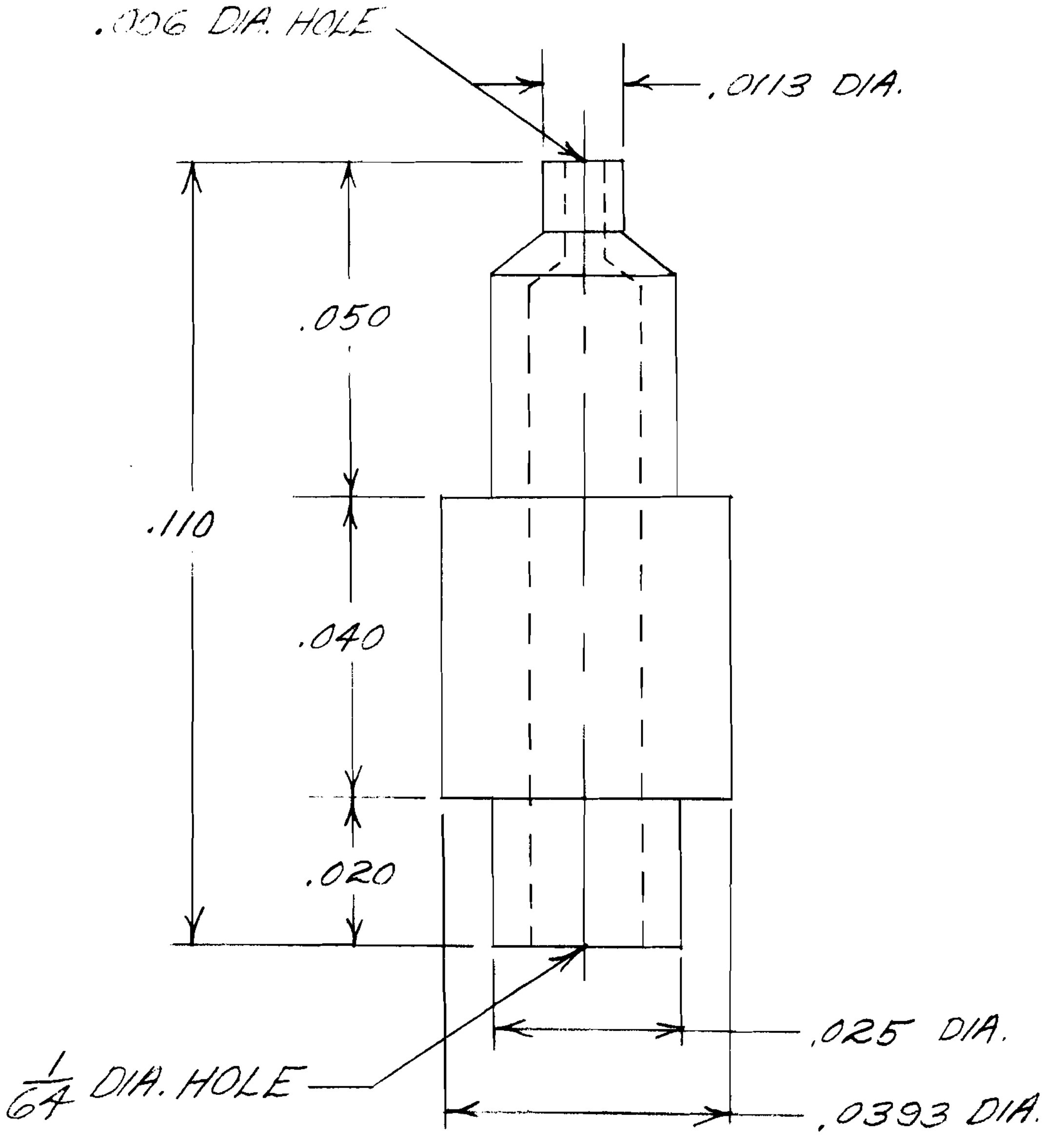


Fig. **12.** Insert orifice

achieved without prohibitive cost. The insert orifice can be purchased directly from a supplier at a very reasonable cost, thereby eliminating the need for development of special machinery to produce these units. Also, the design of the insert orifices is such that it is adaptable to a variety of configurations which would be important in any scale-up of the process.

The crucible/susceptor system, as it evolved in the course of this program, has proven satisfactory. We would not foresee any major changes to this basic system in any future scale-ups of the process.

10. Spooling mechanism

The spooling mechanism is shown in Fig. 13. Here, the as-grown, coated filaments are wound on 12-in. diameter spools, ready for storing or shipping. The spooling mechanism consists of two banks of 13 spools each which are mounted on two drive shafts in the spooler housing. The shafts are driven by a constant-speed ac motor through a gear-and-chain drive train. The motor speed was selected so that the spooling speed would be greater than the pulling speed (i.e., approximately 3 in./min versus 1-1/2 to 2 in./min for pulling) in the absense of constraint. In practice the spooling speed is constrainted to be the same as the pulling speed, since the spooler pulls against the exit side of the belt puller.

Each spool is fitted with an integral slip clutch (Fig. 14) which is installed by inserting the clutch in the hole in the spool and then screwing on the retainer with a spanner wrench to firmly secure the spool to the clutch. The slip clutch consists of a steel shaft bushing, a brass spool bushing, a compression spring, a steel pressure plate, a steel spring retaining collar, and the spool retainer. The steel shaft bushing contains a pin which fits into the keyway in the spooler shaft so that when the unit is operating the steel bushing will revolve at the same speed as the shaft. The brass spool bushing revolves at a slower speed (equivalent to the pulling speed) and is continuously slipping due to the tension in the filaments. The slip torque setting is adjusted by moving the spring-retaining collar in to compress the spring, thereby applying more force to the pressure plate and increasing the friction. The slip torque is set so that there is sufficient force to wind the filaments, yet not override the puller, resulting in a fraction of a pound of tension in the filaments.

The spool/clutch assemblies are mounted in the spooler by sliding them on the drive shafts. These assemblies are positioned in their proper location on the shafts by use of shaft collars which act as stops. A plexiglas plate with bronze bushings is used to support the ends of the shafts.

The filaments are directed from the pulling mechanism to the spooling mechanism through an array of Teflon tubing which starts at the top of the filament machine and terminates in the spooling mechanisms. This arrangement is used to fan out the filament array from a 1-in. wide cluster to approximately a 13-in. span so that, as the filaments exit from the tubing, they are in alignment with their respective spools.

The spooling mechanism is 31-5/8 in. high, 29 in. wide, and 15 in. deep. It is constructed from 1/2-in. thick aluminum plate. When installed on the filament machine frame it is mounted on two brackets which extend from the main baseplate. It is positioned at a 45°

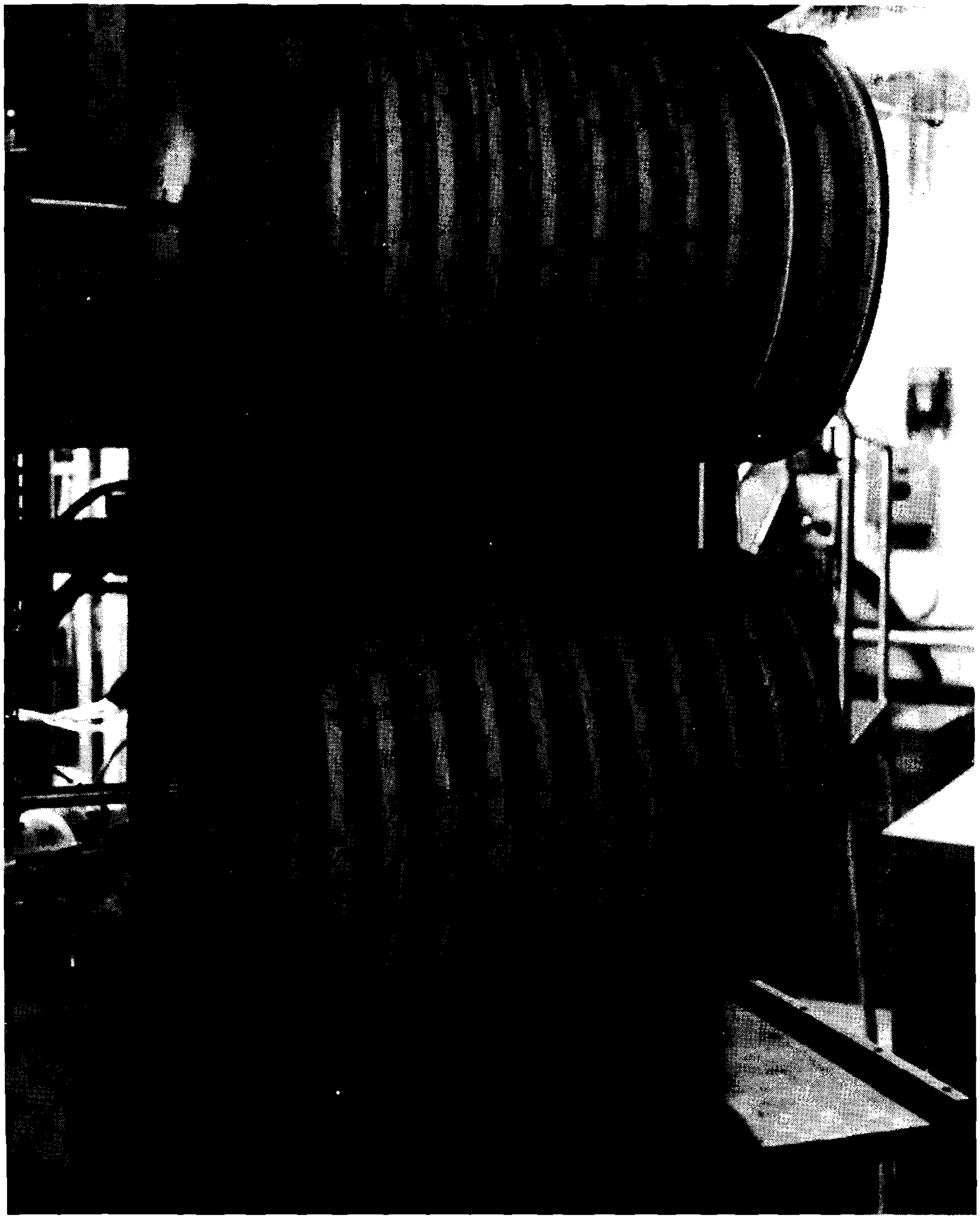


Fig. 13. Spooling mechanism

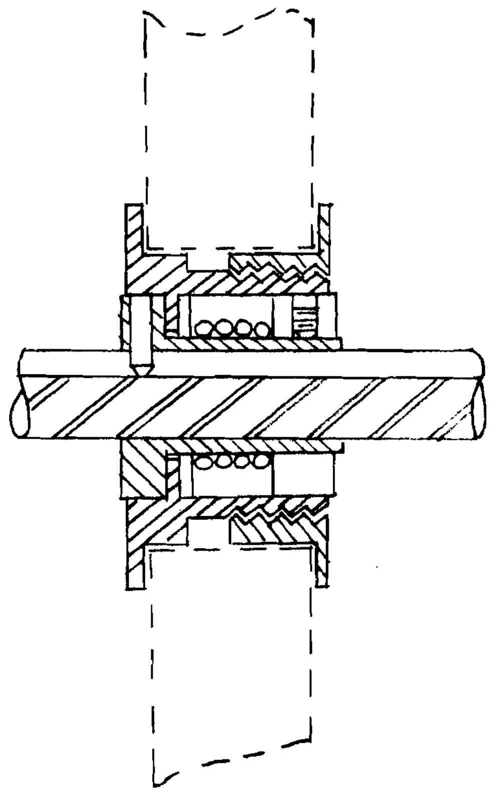


Fig. **14.** Slip clutch assembly

angle to the machine centerline so that the shafts of the spools are parallel to the plane passing through the orifice array. This is necessary so that the filaments will not spin on their axes due to the winding action.

The spooling mechanism, as designed, is considered satisfactory for the collection of 25 filaments. However, for a scaled-up operation a new spooler would be required to reduce the labor involved in attaching the filaments to the spools and loading and unloading the spools. One of the time-consuming aspects involved is the installation and removal of the slip clutch assemblies for each run. A new design spooler would also require a new spool design. The new spool would be thinner, lighter, and have reasonably accurate dimensions. A thinner spool would be required to hold the size of the spooling mechanism down to a reasonable level. A lighter spool would be required to make handling and shipping easier and less expensive. Most likely these requirements could be achieved with a molded plastic spool which could be produced economically on a large quantity basis. Ideally, a larger spooling mechanism would be designed so that the spools could be removed individually. This would require using a peripheral drive (such as a friction roller) instead of an axial drive, as presently used. With this design the slip clutch could be permanently installed in the drive roller. A system such as this would be more complex and expensive, but the savings in operating labor costs would more than justify the higher initial cost.

11. Coating mechanism

From past experience, we have ascertained that even normal handling of sapphire filament can reduce the tensile strength by at least 100,000 psi. This is caused by altering the characteristics of the filament surface by introducing microscopic scratches. For this reason, it was decided that a protective coating should be applied to the filament surface immediately as it left the guide. This coating should be strong enough to protect the filament, be easily removable, and, preferably, be easy to apply. After investigating various materials for this application, it was decided that an acrylic copolymer, Acryloid B72 (Rohm and Haas, Inc.), met our requirements. It provides an extremely tough and durable protective coating, is easy to apply, and can easily be removed with the proper solvent.

The method of application is simply to draw the filament through a pool of Acryloid B72 as it leaves the guide. The Acryloid B72 is diluted with MEK (methyl ethyl ketone) to achieve the proper viscosity required for our operation. Coating thickness is determined mainly by filament growing speed and the viscosity of the coating material. The coating thickness achieved on our test samples was measured to be between 0.00005 and 0.0001 in. thick.

The coater consists of two Teflon sections which, when assembled, form a small chamber which holds the coating solution. At the bottom of the chamber there are 25 slots, 0.016 in. × 0.016 in., spaced 0.040 in. apart, through which the filaments pass upward and through the coating solution. The coater is mounted on the top filament retainer of the pulling mechanism and the slots in the coater are aligned with the slots in the retainer. From the coater the filaments travel upward approximately 10 in. to where they enter the Teflon guide tubes which direct them to the spooling mechanism.

The coater performance has been moderately satisfactory. Some minor problems have occurred with leakage of the coating solution through the slots. While this is messy, it does not cause any major problems. The major problem with this unit is that it is awkward to use in instances where it is necessary to replace seeds.

As noted below in Section II.E., Production Run, the coating unit was not used during the production run. The failure to use this unit, however, was related to the poor growth conditions prevailing at that time. Thus, late delivery of our insert orifices had required that the production run begin with reclaimed setups in poor condition. This and several other difficulties caused both empty filament slots (plugged orifices) and early termination of several filaments in many runs. These conditions, in turn, lead to increased dripping of solution from empty filament slots. In addition, frequent arcing out of the rf generator terminated runs abruptly, requiring re-setup of the machine. For these reasons, it was not convenient to use the coating device during the production run.

D. Process Evaluation

A process for the production of single crystal multiple filament has been established in this program. The process as it now stands is capable of the simultaneous growth of 25 filaments, providing all the operating conditions are perfect or near perfect. The basic requirements necessary to obtain continuous growth of good quality filament and a high yield factor have been established. These requirements are as follows:

1. Growth rate

The upper growth rate limit has been established at 8 ft/hr. Above 8 ft/hr the process is not as stable as required for multiple filament growth and is more subject to filament terminations due to temperature fluctuations in the system. The growth is more stable at lower speeds (e.g., 6 to 7 ft/hr), but this represents a significant decrease in the output and also approaches the point where there is danger of melting the crucible due to the higher operating temperature required. When the growth speed is maintained at 8 ft/hr or slower, the filament is smoother and has less of a beaded effect than when grown at higher speeds.

2. Alignment

The accurate alignment of the filament pulling axis to the vertical axis of the orifice tip is absolutely essential to the continuous growth of good filament. The filament axis must be coaxial to the orifice axis within 0.0015 in. T. I. R. Misalignment exceeding this tolerance will result in a lateral force being exerted on the filament, which eventually will cause bending and termination of growth. With the present design crucible assembly we are capable of meeting the aforementioned tolerance requirements. After considerable testing and evaluation, the filament guide slot dimensions have been finalized at 0.0135 in. $\times$ 0.0135 in. It has been found that these dimensions give us the best compromise between retaining the filaments exactly aligned along their pulling axes while allowing clearance between the slot and the filament so that the filament can line itself up with its respective orifice.

3. Pulling

The pulling action must be smooth and constant with no jerking or vibrations which would cause perturbations at the growth interface. The servo-driven belt puller adequately provides the smooth pulling action and the fine speed adjustment that is required.

4. Vibration isolation

In order to grow good quality filament it is essential that the growth interface be free of vibrations. This has been accomplished by designing the equipment with sufficient mass and rigidity, as well as damping, to reduce the effects of external (to the machine) excitations. The vibrations in the system have been sufficiently minimized as to have no discernable effect on filament growth.

5. Orifice feeding

Proper capillary flow of the molten sapphire to the growth interface is necessary to achieve proper growth. This had been a problem area earlier, but after a series of modifications to the setup it no longer adversely affects our growth operations. The main changes that influenced capillary flow were the introduction of the 3/16-in. thick lid and a reduction in the capillary slot width from 0.040 to 0.025 in. With a new setup there is generally no feeding problem. With subsequent use some orifices stop feeding. However, it was found that the orifices at which growth terminated were those which were hotter than others. A buildup of deposit is noted after long runs on all orifices which affects feeding and/or spreading of liquid. This buildup is accelerated on the hotter orifice and results in decreased filament diameters. Since the growth speed could not be decreased to allow these hot filaments to spread to size without affecting the other filaments, they became progressively smaller and eventually terminate. This problem was overcome by lapping the orifice tips after each run to remove any buildup present and to eliminate any differences in height or diameter and also by grinding down the bottom of the radiation shield so that the position of the orifice tips relative to the shield is not changed. Some minor degree of misalignment can be tolerated by the clearance in the guide slots, but if this tolerance is exceeded the filaments involved will terminate growth after pulling is initiated because the bends reproduce and became larger.

Straight seeds, when used in the present system, are concentric to the orifice tip axis within 0.0015 in. after alignment. Filaments not initially straight will continue bending in the manner described and present the worst situation because they can bump into adjacent filaments causing them to start to bend and eventually terminate. Straightness, therefore, is the overriding requirement for seed stock in multiple filament growth. Smoothness and even the diamter size, although important, are secondary to straightness. Subsequently, it was found that this reconditioning of the setups after each run reduced filament growth terminations to an acceptable frequency.

6. Chamber atmosphere

Chamber contamination due to air entering the system has been virtually eliminated. However, there is a progressive accumulation of deposits in the furnace chamber during operation. Samples of these deposits were removed and sent to a testing laboratory for spectrographic analysis. The laboratory report showed that the deposits consisted mainly of aluminum. This indicates that the Al_2O_3 charge is vaporizing and precipitating out on the cooler parts of the furnace chamber. These deposits appear to have no adverse effects on the growth operations, and runs of up to 20 hr have been attained. However, the deposits are undesirable in that the system requires extensive cleaning after each run and also that the deposits tend to accumulate on the growing filaments. It is probable that with further development, the Al_2O_3 vaporization could be reduced or minimized. This might be accomplished by raising the pressure in the furnace chamber to reduce vaporization or by modifying the crucible assembly such that the melt temperature is reduced.

7. Filament seeds

It is essential that the seeds used to initiate a growth run be straight for two reasons. First, unless all the seeds are straight, the initial alignment will be off in that the seed ends, dropped through the guide, will not line up with their respective orifices. Second, if seeding is done with a nonstraight seed, then the characteristic behavior is that the bend in the seed reproduces itself in exaggerated fashion, over and over, until surface tension will no longer hold the growing filament on the orifice.

Seed crystal orientation should be with the c-axis parallel to the filament axis within 1°. This requirement is related to the straightness criterion in that apparently-straight seeds which are misaligned are more likely to develop bends as they continue to grow.

8. Temperature regulation

After growth has been initiated, the most important factor in maintaining the proper growth situation is temperature regulation. During the production run, it was observed that a drop of 5°C in the operating temperature, as measured by the Ircon Optical pyrometer on the primary radiation shield, was sufficient to cause the filaments to grow cold and, as a result, terminate growth. Temperature sensitivity can be compensated for to some extent by growing the filaments a little faster and thinner for the particular temperature setting and therefore allowing more latitude for temperature drop before growth is affected. This is undesirable, however, because now there is reduced latitude for upside temperature fluctuations and because other aspects affecting stable growth are deleteriously altered under hot conditions.

In retrospective analysis it appears that the insert orifice tips are extremely susceptible to temperature fluctuations and also that their thermal reaction time is faster than the other parts of the setup. For this reason a remote sighting location for the optical pyrometer (such as the primary radiation shield) does not provide adequate temperature control, since a small change in temperature on the shield may be reflecting a significantly larger temperature change at the orifice array. With the present Ircon optical pyrometer temperature sensing system, it is not possible to sight on the orifice tips or growth interface.

During the production run the best results were obtained using the manual control, with the operator making the necessary power level and speed adjustments to maintain the filament at the proper growth temperature. In this way, temperature corrections are based on the actual conditions at the orifices rather than on a related condition somewhere else which may be changing independent of the growth conditions. However, from our production growth experience it is also evident that a completely automatic control system is feasible with further development effort.

Serious consideration would be given to a two-part control system whereby the basic temperature control would be provided by a unit similar to the present design (which is satisfactory for this purpose) and a second component to the system would provide fast response by making minor adjustments in the growth speed. In this way the basic operating temperature would be maintained by the primary unit, and the fluctuations in the system would be compensated for by the second unit which would adjust the growth speed. A sensor would be required to provide the fast response unit with the necessary input data. A possible device for this purpose could be an electro-optical sensor which would monitor the diameter of a key filament and supply an output signal in proportion to the magnitude of increase or decrease in diameter. This signal would be used to provide commands to the pulling mechanism control unit to make the necessary speed corrections.

9. Orifice tip diameter

Analyzing the results of the production run indicates that a slight increase in the orifice tip diameter would probably result in more filament meeting the specified diameter tolerance of 0.010 ± 0.0005 in. The present tip size is 0.0113 in. and the proposed change in diameter would be to 0.0115 in., to be verified by testing.

E. Production Run

The goal of the production run was to operate the machine daily for one month to produce 20,000 ft of 0.010 in. diameter filament. This goal can therefore be broken down into a performance goal of 22 individual runs, each of which would require the production of 910 ft of filament. Compared to these requirements, it was shown that operation of the machine under ideal but realizable conditions resulted in a production capability of close to 1200 ft per day. This capacity is reached when 20 or more filaments grow during a run sustained for 6 to 8 hr.

In fact, although sustained growth runs of 20 or more filaments were often achieved, the average number of filaments successfully propagated per run was less than 20. This circumstance is explained by a set of extremely unfavorable conditions existing at the beginning of the production run, together with the fact that we did not place emphasis, at that time, upon attaining the full capacity of the machine in terms of the number of filaments propagated.

The production run began on schedule after the end of the twenty-first contract month. Initially, the conditions for growth were poor due to late delivery of insert orifices needed to prepare new setups, malfunctioning of the rf generator, and receipt of several improperly fabricated chambers from the vendor. We therefore began using old setups. These setups were unsatisfactory due to geometrical deficiencies resulting from repeated lapping of their tips, and a reduced number of functioning orifices due to a few tips which had become clogged. The former was particuarly serious inasmuch as the reduced height of the orifice tips apparently interacted with the heat shielding to produce a temperature gradient across the orifice tip array which made it difficult to maintain the growth of a large number of filaments.

The malfunction of the rf generator was the most serious problem and took the longest time to solve. The malfunction was manifested in two ways. First, there was a high incidence of abrupt and complete failures of the set which would terminate an entire run. But, worse than that, it was found that the power output of the machine tended to fluctuate so that some of the filaments would not pass the guide and would eventually terminate. All of these problems were finally traced to the ingestion of carbon fiber particles from the felt insulation used in the plate setup in the same laboratory. The rf set was thoroughly reworked and cleaned, and filters were installed in its air-intake area.

Since the production run began, the rate of filament production has shown a steady increase. The increased rate was, in a large part, possible by the eventual receipt and use of new orifice arrays and by the proper refinement of seeding techniques. During the first three months after the production run began, 22,000 ft of filament were grown. The average per run increased from only 392 ft during the first month to 595 ft during the third. At the same time, the number of filaments successfully propagated increased from 12 to 17. During the third month, 10 of 21 runs carried out produced 8436 ft of filament, or an average of 843.6 ft per run. During the same 10 runs, the number of filaments propagated varied from 17 to 22 and averaged 19.

The increased rate of production during the third month resulted from use of newer setups and improved operator efficiency, particularly during the seeding operation. Satisfactory repair of the rf set to eliminate the abrupt shutdowns and power fluctuations had not been effected during this time and were the primary cause of the poor performance during half of the third month's runs. The repairs were finally completed, but not in time to generate production statistics.

In addition to the power supply and the use of old setups which had affected performance, various other factors more endemic to the system itself were brought under control to improve the production rate. One of the most significant aspects of the continuous long-term growth of filament is the choice of seeds and the actual seeding operation. Although we have described these factors elsewhere, it is worth reiteration to state that it is absolutely essential that the seeds meet a minimum tolerance for both straightness and accuracy of crystallographic orientation in order to assure reasonable yield in terms of the number of filaments which grow throughout a given run. Use of bent seeds makes seeding itself uncertain, while use of either bent or off-orientation seeds results in the propagation of a succession of worsening bends which eventually cause termination. It is also true that termination of one filament by this propagating bend mechanism is often catastrophic because in terminating, the loose end often whips past other filaments and causes other terminations at the same time.

Power fluctuations in the rf output caused the loss of filaments by a mechanism related to this straightness criteria. The momentary reduction of rf power lowers the crucible temperature and, as a result, decreases the height of the liquid film on the orifice tips from which the filament growth takes place. It is known that when the growth becomes cold (i.e., liquid film thickness decreases), the crystal being grown shows a tendency to bend in proportion to the reduction in film thickness. As noted above, any bends thus formed, if not immediately catastrophic, increase in severity as the first bend enters the guide. Thus, power fluctuations cause bending, subsequent filament termination and, therefore, decreased production rate. During growth runs with the rf set in good condtion, this problem is eliminated.

Filament was characterized and classified into five categories: A, B, C, D, and remaining material. The classifications are as follows:

A Material	-	Both ends of the filament 400,000 psi or greater and the average of both ends greater than 400,000 psi
B Material	-	One end 400,000 psi or greater and the average of both ends less than 400,000 psi
C Material	-	Both ends 350,000 psi or greater and average of both ends greater than 350,000 psi
D Material	-	Both ends 300,000 psi or greater
Remainder	-	All other material grown

Filament production for the months January, February, March and April totaled 30,305 ft Production figures for the respective months are as follows:

	No. of Product Runs	Total footage grown
January	19	6,275 ft
February	8	3,396 ft
March	27	12,419 ft
April	13	8,215 ft

Of the 30,305 ft of filament grown, 21,081 ft have been characterized as A, B, C, or D material. The breakdown is as follows:

A Material:

0.007 dia	0.008 dia	0.009 dia	0.010 dia	0.011 dia
189 ft	504 ft	2,148 ft	2,976 ft	107 ft

Total: 5,924 ft

B Material:

0.007 dia	0.008 dia	0.009 dia	0.010 dia	0.011 dia
39 ft	268 ft	1,833 ft	3,266 ft	223 ft

Total: 5,629 ft

C Material:

0.007 dia	0.008 dia	0.009 dia	0.010 dia	0.011 dia
112 ft	343 ft	1,792 ft	2,574 ft	243 ft

Total: 5,064 ft

D Material:

0.007 dia	0.008 dia	0.009 dia	0.010 dia	0.011 dia
142 ft	315 ft	1,328 ft	2,143 ft	536 ft

Total: 4,464 ft

Remainder: 9,224 ft

NOTE: Diameters (± 0.0005) measured at finished ends

In regard to the quality of the filament, several important factors should be considered. Due to the previously mentioned difficulties it was found to be awkward to use the coating unit, and consequently the filament was not coated. Therefore, the filament was vulnerable to handling damage and the resultant loss in strength. In addition, the test specimens are taken from the beginning and the end of each length which is the worst case for providing a representative sample of the entire filament. Also, when a new setup is used the first run always produces lower-than-normal strength material. The exact reason for this is not known, although it could probably be assumed that the molybdenum components contain some impurities which are carried off in the initial growth. Finally, the tensile testing itself results in lower-than-actual readings. This involves the selection of the test specimen, the adverse effects of beading the ends of the specimens, and small misalignment in the test fixture. Since the testing will never produce a higher-than-actual strength reading but can produce lower readings, then, in view of the large number of samples tested, it is likely that some of the test results represent lower-than-actual filament strengths. These negative factors are presented only so that the reader can consider all aspects of the situation in analyzing the results of the production run.

A breakdown of the A, B, C, and D material (total footage 21,081 ft) as to their diameters (measured at the finished ends) is as follows:

0.007 dia	0.008 dia	0.009 dia	0.010 dia	0.011 dia
482 ft	1,430 ft	7,101 ft	10,959 ft	1109 ft
2.3%	6.8%	33.7%	52%	5.3%

In analyzing these results, it should be noted that nearly 90% of the material grown was 0.010 in. within ± 0.001 in. Also, most of the material which is not 0.010 ± 0.0005 is on the low side. This would indicate that a minor increase in the orifice tip diameter could result in substantially more filament being within the specified tolerance.

During the growth operation the proper filament diameter is obtained by the machine operator making a judgment based on visual observation of the growth interface under suitable magnification. The operator compares the growing filament diameter to the orifice tip diameter and makes the necessary growth speed adjustments to achieve the proper filament diameter. A certain degree of experience is required to estimate the filament diameter using this method. During the production run a progressive improvement in maintaining the filament diameters, within the specified tolerances, was observed. This can be attributed, for the most part, to increased operator skill. Another important factor which contributed to maintaining proper diameters was the final design change in the radiation shield, which virtually eliminated the thermal gradient across the orifice array.

In summary, although the production run was far more prolonged than had been anticipated, or desired, it has been successful in that it has provided necessary skill in and understanding of the operation of the machine, and has demonstrated the production rate capability which was the goal of the contract. Causes for the poor performance during most of the production run have been identified and eliminated, and we are therefore confident that the existing equipment is fully satisfactory and capable of sustained growth of 20 to 25 filaments simultaneously.

F. Filament Characterization

All filaments produced during production were characterized as to diameter and tensile strength and were given a visual examination to check surface characteristics. A test sample was taken from each end of the filament so that tensile strength values could be obtained for the start and the end of each filament in each run.

This procedure of checking each filament was adopted because the early yield during the production run was very low. However, a statistical test procedure was developed and shown accurately to characterize the average and variability of each run as a whole by testing not more than 24 samples randomly picked from each complete run.

A special test fixture was developed to insure accurate alignment of the test specimen in the tensile test as well as to simplify the test procedure. The fixture is designed to be used in conjunction with test specimens which are potted in small aluminum channels. The latter are machined to fit in the test fixture in a set position. A separate potting fixture is used to mount the specimens in the holders. Each potting fixture can accommodate 12 specimens and is designed to assure accurate alignment of the specimens in the holder. Prior to potting, each specimen is "beaded" on each end using an oxy-hydrogen flame. These beads provide additional holding power and prevent the filaments from slipping in the grip. The potting agent used is Eccobond 285 with catalyst 24 LV from Emerson & Cuming, Inc., Canton, Massachusetts. This epoxy provides the high-strength adhesive bond necessary and also has a fast curing time.

The test fixture fits into the Instron Model TTDL tensile testing machine and replaces the standard gripping jaws. The test specimens, in their holders, are placed in the fixture and the machine is operated to perform the test. This machine has incorporated a strip chart recorder which provides a graphic record of the force required to fracture each test specimen. After fracture the diameter of the specimen is measured at the fracture point and this information, along with the specimen code number, is entered in the chart record. At the completion of testing, the specimen strengths are calculated from the data recorded on the strip chart and this information is then logged in a "Filament Test Report" for the particular production run involved. Also included in this report are the lengths of the individual filaments.

The tensile testing procedure has proved adequate for our purposes. The holders are reused by removing the epoxy using a stripping agent (Eccostrip 93). In a full production operation, testing would not be performed on a 1009 basis but, instead, a random sampling technique would be used to determine the average tensile strength of the entire run.

The modulus of elasticity for c-axis oriented sapphire filament has been established at 67.2×10^6 psi. This was accomplished under United States Air Force Contract No. F33615-68-C-1126, and the procedure used to determine the modulus is presented in detail in Technical Report AFML-TR-69-102, "Continuous Sapphire Filament Production," April, 1969.

G. Quality Control

To insure that sapphire filament quality standards are maintained, certain quality control procedures have been implemented. These procedures are as follows:

1. Raw material purity

The source material for sapphire filaments is scrap white verneuil-grown sapphire boules. These boules are processed here at Tyco to achieve the proper mesh size particles used in the filament growth process. A chemical analysis of the source material demonstrates the relatively high purity of these boules.

Chemical Analysis of Starting Material

Aluminum, %	$>$ 10
Molybdenum, %	0.003 - 0.0003
Iron, %	$<$ 0.0001
Silicon, %	$<$ 0.0003
Copper, %	$<$ 0.0001
Magnesium, %	$<$ 0.0001
Beryllium, %	$<$ 0.0001
Sodium, %	$<$ 0.0001
Manganese, %	$<$ 0.0001
Phosphorous, %	ND

The boules are thermally shocked by heating them to 1000°C and quenching them in water to crack them as the first step in the process. They are subsequently mechanically crushed and screened to obtain sapphire particles of -10 +30 mesh size. During the preparation of the material, measures are taken to insure that purity is maintained. The basic procedure is as follows:

a. Visual inspection

Initially, all boules are inspected visually. An inspector examines the boules and rejects those which are discolored.

b. Chemical etching

After the material has been pulverized to achieve the desired mesh size particles, it is subjected to the following chemical etching procedure to remove any impurities which may have been imparted to the material as a result of the crushing operation.

Chemical Etching

- Place approximately 1/2-in. thick layer of sieved material in a polypropylene or Teflon pan and cover it with aqua regia (3 parts HCl + 1 part HNO_3).
- Allow the material to etch for about 4 hr.
- Remove the acid and rinse with water several times.
- Again cover the material with acid and allow it to remain covered for 24 hr.
- Remove the acid and rinse the material liberally with water.
- Rinse the material with distilled water 5 or more times and check for the presence of acid using pH paper.
- When the pH of the water in the material is the same as the pH of the distilled water, then spread the material out on aluminum foil and dry it completely in an oven at 80 to 100°C.
- When completely dry, allow it to cool to room temperature and store the cleaned material in polypropylene or Teflon containers for future use.

2. Crystal orientation

To insure that the filament produced has the proper orientation, it is necessary that the seed filaments be such that their filament axis is parallel to the c-axis within 1°. The seeds are checked for proper orientation using an X-ray photograph to observe the crystal structure. The procedure is to mount a sample of the seed in a goniometer and align the seed axis so that when the unit is installed in the X-ray machine the axis will be normal to the X-ray beam. Then a Laué back reflection photograph is taken using a Polaroid camera. The photograph is analyzed to determine the deviation from the c-axis.

Those seeds exceeding the specified deviation tolerance are rejected.

3. Cross-section geometry

The circular cross section of the filaments, by the basic nature of the EFG process, is defined by the shape of the orifice tips. Since the insert orifice tips are generated by a turning operation, they are well within tolerance requirements for roundness. After drilling and prior to final installation in the orifice mount, the tips are inspected under high magnification and those units not meeting the specified tolerances are rejected. The filament cross section is checked by measuring the diameter at three different positions on the circumference at the same linear location.

4. Surface coating uniformity

The surface coating uniformity is checked by using a micrometer to measure the diameter of the coated filament at several locations along the length of the filament. The coating can be burned off or removed with a solvent (MEK) and the original diameter of the filament measured to arrive at the coating thickness. With the type of coating material (Acryloid B72) currently used, the coating thickness is on the order of less than 0.0001 in. thick and, consequently, the coating thickness is not as critical as originally anticipated.

H. System Operating Procedure

This section covers the operating procedures used to grow multiple sapphire filaments.

1. Crucible/susceptor assembly preparation

Prior to their first growth operation, the graphite components (i.e., the susceptor, pedestal mount, and the graphite felt insulation) are baked out in order to remove any impurities present in these components. This is accomplished by installing these components in the furnace chamber, in their normal operating positions, and bringing the temperature slightly above the normal operating temperature. Considerable smoke will be produced as the impurities burn off, and the absence of smoke can be used as an indication that the bake-out process is completed. This bake-out procedure is only necessary once for each graphite component. The inside of the furnace chamber must be cleaned out after every bake-out operation to remove the impurities from the inside walls.

The entire crucible assembly, including the orifice array, the radiation shields, the crucible liner, and all of the various parts used in this assembly, must be thoroughly cleaned prior to the growth operation. This is accomplished by placing the parts in a beaker of acetone and using an ultrasonic cleaning unit to achieve the high degree of cleanliness required. Three to five minutes in the ultrasonic cleaner are sufficient.

Special attention is given to the orifice array, especially the orifices themselves. After the array is removed from the ultrasonic cleaner, a light stream of air may be used to blow out any residual acetone, chips, particles, etc. At this time the orifices are checked individually under a microscope or optical comparator to verify that the feed holes are clear and free of obstructions. If any of the orifices are not clear, the aforementioned procedure is repeated. After the components have been cleaned they are handled only with nylon or plastic gloves by the operator. The orifice array is also checked to see that all of the orifices are seated properly.

After cleaning the crucible assembly, it is assembled in its normal operating arrangement and mounted in the susceptor. The crucible/susceptor assembly is then installed in the furnace chamber.

The tungsten locating pin and the three support pins are installed in the bottom of the crucible. The crucible is then put into the susceptor. The locating pin slides through a pilot hole in the base of the susceptor and maintains the crucible in its proper position in the susceptor. The crucible sits on the support pins.

The susceptor, with the crucible assembly inside, is then installed in the pedestal mount which has been bolted to the mounting block on the movable platform. The susceptor is installed by pushing its mounting shank into the hole in the top of the pedestal mount. Positioning of the orifice array is accomplished by turning the susceptor until the array is at a 45° angle to the front to back horizontal centerline of the filament machine. This can be checked by moving the crucible/susceptor assembly up into operating position and using two filament seeds in the extreme slots of the guide. Next, the secondary (tungsten) radiation shields are placed in their proper location on top of the susceptor and the graphite felt is installed. The crucible/susceptor assembly is raised into its operating position and the platform is locked in place by the two hand-turned screws in the main baseplate.

2. Seed/installation

The preselected seeds are installed in the filament machine in the following manner.

a. All 25 filament seeds are inserted into the plastic block in the top plate of the machine and pushed up into their respective Teflon tubes.

b. The seeds are fed into their respective holes in the top retainer of the pulling mechanism. Tweezers may be used to do this.

c. The seeds are installed in the coating mechanism applicator unit. This is accomplished by loosening the two wing nuts and removing the front plate of the unit. Next, the seeds are positioned in their respective slots in the applicator unit and the front plate is put back on, thereby retaining the seeds in the slots.

d. Now, the seeds are inserted in their respective slots in the guide and allowed to enter the furnace chamber. At this point the guide-positioning mechanism should be adjusted so that the seeds will all come down and rest on the primary radiation shield. This is to insure that all seed ends are in the same alignment plane.

e. At this time the pulling mechanism pressure plates are actuated so that the filament seeds are secured between the belts.

f. The pulling mechanism is activated to raise the seeds. The seed array is observed during the pulling action to check that everything is functioning properly, i.e., that the spacing between the filaments remains constant and that they enter and proceed into the guide tubing assembly. The seeds are not raised beyond the point where they would leave the guide.

g. The direction of the pulling mechanism is reversed to slowly lower the seeds to just above the shield. The seed array is observed through the microscope to note that all of the edges are still on the same plane. If not, the alignment procedure is repeated, the pulling mechanism backing plane pressure is increased slightly, and the checking procedure is repeated.

h. The guide-positioning mechanism is readjusted so the seeds are positioned above the orifices. The seed installation is now complete.

3. Start-up procedure

At this point the growth operation is ready to be started. The start-up procedure is as follows:

a. The gas flow to the feeding mechanism and the furnace chamber is turned on and the flow rates are checked.

b. The rf generator is energized. When the ready light comes on the coil is energized.

c. The unit is brought up to operating temperature slowly by raising the power setting in a series of steps. The time to raise the unit to full operating temperature should be twenty minutes, and the power setting should be raised in a series of steps allowing a stabilization period at each step.

d. The continuous feeding mechanism is turned on to start the charging operation.

e. Now the automatic temperature control is actuated and the optical pyrometer is lighted. The meter on the temperature control panel is nulled to observe the proper operating temperature.

After completing the start-up operation, the system is ready for the seeding and the start of growth operations.

During the seeding operation it is desirable to maintain the temperature slightly above (i.e., approx. 20 to 30°C) the normal temperature. Using the pulling mechanism, set at slow travel, the seed array is brought down to a point just above the orifice array and the necessary corrections are made using the guide positioning mechanism, to bring the seeds in alignment with the orifices. Alignment is checked by viewing the array through the other viewport and making further corrections, if necessary. The seeds are then brought down, slowly, to just touch the orifices, and a final check of alignment is made. Pulling speed is then set at 2 in./min and growth is initiated, maintained for 1/8 in., and then stopped. A visual inspection of the newly-grown sections will determine if any further alignment corrections are necessary. If corrections are necessary, the junction is remelted by slowly lowering into the orifices and then pulling the seeds free by raising them at high pulling speeds. The necessary alignment corrections are made and the seeding repeated. When no further corrections are necessary, the pulling speed is set at the proper rate (i.e., 1-1/2 in./min) and growth is allowed to proceed.

During the initial growth an observation of the filament diameter will indicate whether a temperature change is necessary. The filament diameter should be slightly smaller than the orifice. The meniscus should be low and steady. If the seeding was correct and the junctions not overly large, the filaments will be steady as the junctions pass through the guide and no difficulties will arise. Until the junctions are through the restainer above the puller, a close watch should be kept on all of the orifices. In the event any mishap occurs, fast reaction can avoid termination of the run. A mishap could occur if any of the filaments become too cold. In this case the filament could become distorted and bind up in the guide or damage to the orifice could occur. Close observation of the meniscus can be used to determine if the filament is running cold and the situation can be corrected by adjusting the temperature setting. If the temperature becomes too hot, the filament diameters will decrease until the filaments are pulled free. This also can be avoided by close observation and making the necessary temperature setting adjustment.

In any case, it is obvious that during initial stages of growth, while the temperature is equilibrating at the correct growth set point and the junctions are passing through the guide and puller, close observation is required. Once this critical period is passed and all parameters are correct, the growth proceeds undisturbed.

The filament growth process in itself is self-stabilizing or self-correcting once the system reaches thermal equilibrium at the growth interface. That is, once the proper settings for temperature and pulling speed have been set and good growth is underway, no further corrections or adjustments should be required. However, the process should be monitored periodically to verify that everything is functioning properly.

As a check on filament quality, the growing filament should be observed both in the furnace chamber and also as it leaves the guide. The filament should be smooth and not exhibit any beaded appearance, which would indicate a constantly varying diameter. This can be checked by visual observation or by feeling it. Also, as the newly grown filament (i.e., not the seed stock) emerges from the Teflon guide tubes, test samples from each orifice should be removed and subjected to characterization tests to verify that the filament quality meets the requirements.

I. System Scale-up Capability

The figure of 25 filaments was originally selected only as a basis for demonstrating the feasibility of developing a sapphire multiple filament growth process. It was assumed that if 25 filaments could be grown successfully with a reasonable yield factor and the equipment demonstrated to perform satisfactorily, then the system could be scaled up to grow larger quantities of filaments simultaneously. The program has, in fact, demonstrated the capability for multiple filament growth and also the definite potential for scaling up the operation to a larger number of filaments. A reasonable quantity for scale-up could be of the order of 200 filaments simultaneously. This quantity is selected based on the following conclusions.

The primary reason for any scale-up of the process would be to reduce the cost of filament a significant amount. Consequently, the larger quantity of filaments which could be grown simultaneously using the same machine, same power unit, etc., would lower the operating cost and lower the cost of the basic equipment. However, there is an upper practical limit to the quantity of filaments which could be produced simultaneously in a single system on a sound economic basis. Attempting to grow an unreasonably large quantity would certainly add greatly to the complexity (and consequently to the cost) of the equipment, as well as the growth problems, beyond reasonable limits.

On the basis, the quantity of 200 filaments was derived as a figure which represents a significant increase over the present capability of 25 filaments and yet is within practical design limits. A system capable of growing 200 filaments at 8 ft/hr, with an 80% yield factor, would produce 1,280 ft of filament per hr, or 10,240 ft in an 8-hr period of continuous growth. Assuming one operator could monitor three machines, this would represent 30,720 ft of filament per 8-hr day/operator, or approximately 4 lb of filament. Of course, additional labor would be required for setting up the machine, removing the spools, testing, and calculating the filament strength and various other support services that are required to maintain the production process. However, it can be readily seen that on the basis of producing 200 filaments simultaneously, the cost of filament would be reduced to competitive commercial levels.

Consideration of the design and development aspects of scaling up to 200 filaments indicates that the problems involved are within reasonable bounds and pose no "beyond the state of the art" research and engineering. The feasibility of multiple filament growth has already been demonstrated on this program and what needs to be considered are the difficulties in scaling up to a larger quantity.

First, it is felt that 200 filaments could easily be accommodated by a 3-in. O. D. crucible and possibly even a slightly smaller crucible. The orifice array would perhaps be five linear rows of 40 orifices or one or two circular rings of orifices. The actual layout of the array, at this point, cannot be determined, but sufficient experience has been acquired on this program to say that a 3-in. O. D. crucible would be adequate. This means that the furnace chamber size would not be much larger than our present unit (i.e., a 125-mm O. D. chamber versus the present 100-mm O. D. chamber). As a result, it is certain that the same size (20 KW) power source could be utilized. The increase in electrical power requirements to grow 200 filaments versus the 25 filaments would not be significant or greatly increase the operating costs. Further, the cost of the crucible, susceptor, and the various smaller components in the setup would not increase significantly over our present costs. The largest cost increase in the setup would be the cost of the insert orifices due to the increase in quantity from 25 to 200. Presently, the cost is $1.35 each in lots of 500 for the insert orifices less the 0.005-in. dia feed hole, which is generated here at Tyco. Quotes from another vendor (Vallorbs Jewel Co., Lancaster, Pa.) list the cost for 1000 units at $.75 each complete, including the feed hole. Using 200 orifices per setup would probably entail purchasing minimum lots of 5000 units, which should reduce the orifice cost even further. Consequently, the increase in scaling up from 25 to 200 filaments would not be a factor of 8, but rather 2 or 3 times the cost of the present setup produced in a reasonable quantity.

The subsystem most affected by the scale-up would be the pulling mechanism. Whether to use a belt puller similar to the present design or some system with individual filament drives can only be determined in the development of the system. Individual drives certainly would be more complex and expensive yet would offer much better control in that adjustments could be applied to individual filaments rather than to a group or groups of filaments. Of course, the type of pulling system selected would, in a large part, determine the frame design, depending on the size of the unit mounting arrangement involved. Regardless of the type of system required, the development is within the area of basic electro-mechanical design and poses no outstanding difficulties.

Guidance would be accomplished using the rigid guide concept, with the guide design matched to the design of the orifice array. In fact, the design of the guide would influence the design of the array as well as the reciprocal. Closer tolerance of slot spacing would be required and care would have to be taken to eliminate any tolerance buildup.

Spooling and coating would be accomplished using the same basic design approach as presently used. Certainly a more sophisticated spooler would be required to reduce labor costs in removing spooled filament and installing new spools. Counters should be installed to count the footage of the individual filaments to eliminate the present method of rewinding the individual filaments to determine the lengths.

Continuous melt replenishment would be accomplished using the same type of feeding mechanism as the present design. As mentioned previously, a more precise (and sophisticated) temperature control system would be required, especially in the growth of 200 filaments. Certain problems exist in this area. However, now that the problem is well defined, it is obvious that the technology for the solution is known.

Thus, evaluation of the individual components in the 25-filament process has demonstrated that the design concept is basically satisfactory. Various residual problem areas have been defined. It is proposed that further scale-up of the process to 200 filaments would result in greatly reduced manufacturing costs. The scaled-up machine would not entail complications which would obviate the reduction of manufacturing costs.

J. Summary

A multiple filament machine for the simultaneous growth of 25 sapphire filaments has been designed and built. At the conclusion of the program, an extended production run was carried out which demonstrated the production rate capability, which was the major goal of the program, could be obtained.

Detailed analysis of the capability or function of each of the subsystems and materials used in the process has been described. It was shown that details of particular components could further be improved to increase the production rate and yield beyond the goals of this program. This detailed analysis has demonstrated that the multi-filament process is feasible and has further suggested that an additional (8 times) scale-up to 200 filaments is feasible without novel additions. This scale-up would make possible an additional, substantial reduction in filament cost.

III. SAPPHIRE PLATE GROWTH PROCESS

A. Introduction to Section III

This section of the report covers the sapphire plate growth program. It reviews the design and assembly of the plate growth apparatus with particular emphasis on the concept for the heating system, which is essentially new as applied to sapphire growth by EFG. It also reviews the progress made in the growth of large sapphire plates and discusses the various problem areas encountered.

B. Program Objectives

The objective of this section of the program, sapphire plate growth process, was to design and construct an apparatus to grow sapphire plate 12 in. × 12 in. × 3/8 to 3/4 in. thick for transparent ceramic armor. The program was broken down into Phase I and Phase II, with each phase further broken down into smaller increments or tasks.

The objectives in Phase I were to design and construct the various components necessary for the assembly of a complete sapphire plate growth machine. Phase I was separated into several key tasks: (1) heating system, (2) furnace enclosure, (3) vacuum system, (4) crucible fabrication, (5) orifice fabrication, (6) continuous feeding, (7) temperature control, (8) annealing system, (9) pulling mechanism, (10) surface finishing, and (11) assembly and operational instructions. The Phase I objectives were completely and successfully completed.

Phase II of the program developed two problems that were not completely solved within the program time limits: (1) sapphire crystal clarity and (2) sapphire crystal cracking. The objectives successfully completed were: (1) machine assembly, (2) crucible fabrication, (3) orifice fabrication, (4) surface finishing, and (5) optimization. The lack of clarity was caused by vertical strings of small voids located over the capillary openings which appeared in both subscale and full-scale plates. The scale-up from the intermediate to the full size resulted in additional problem areas, particularly in cracking, but also in thermal distribution and supporting components. The problems in the supporting components, such as cover and shield over the crucible assembly, graphite cover, and thermal insulation were solved.

C. Equipment

Past work at Tyco on the growth of sapphire plates was carried out in equipment which basically represents a size scale-up of the basic furnace and crucible assembly such as that described for the growth of filament in Section II of this report. Thus the setup consisted of a molybdenum orifice with molybdenum or tungsten heat shields. The melt was contained in a tungsten crucible * inside a carbon susceptor. This setup was supported within a double-wall quartz, water cooled chamber and heated with a 50 KW 450 KC rf generator. Growth of approximately 2- to 3-in. wide plates was carried out in this way. The problems encountered in this system, however, strongly suggested that further scale-up of that approach was not practical and that a more flexible system for the growth of plate could be built along an essentially different approach.

In this section we describe the equipment which we designed and built for the growth of a 12-in. sapphire plate. This equipment has proven extremely reliable and represents a viable approach to the growth of large plates.

1. Heating system

The method chosen to obtain a temperature in excess of 2050°C to melt the sapphire charge was induction heating and was derived from the successful basic approach of growing sapphire plates up to 3 in. wide. However, the induction heating equipment for the scale-up differs in size and frequency from the laboratory equipment used for the smaller size plates.

The considerations in choosing the power generator source included the mass of material to be heated, the penetration depth obtainable in the susceptor by different rf frequencies, and the throwing power, or coupling capability, which is also a function of the frequency of the rf supply. The larger size sapphire plates require a larger susceptor, crucible, and supporting parts which increase the mass of material to be heated. The greater the mass of material to be heated, the greater the depth of penetration into the susceptor required for uniform heating. Fig. 15 illustrates the depth of penetration at the various frequencies. The initial laboratory work used a 375 to 450 KC power source which has a depth of penetration in a graphite susceptor of 0.080 in. In contrast, in the lower range of frequencies represented by motor generators, penetrations in the range of 0.4 to over 2 in. are readily attained, as shown in Fig. 15. It is of advantage to use these larger penetrations in the present instance because use of thicker, more durable susceptor components can be allowed by so doing.

*Tungsten crucibles were used here to prevent reaction with the carbon susceptor. When molybdenum was used, even in a smaller laboratory plate growth furnace, the increased susceptor temperature required to heat the much larger charge frequently led to reaction between crucible and susceptor.

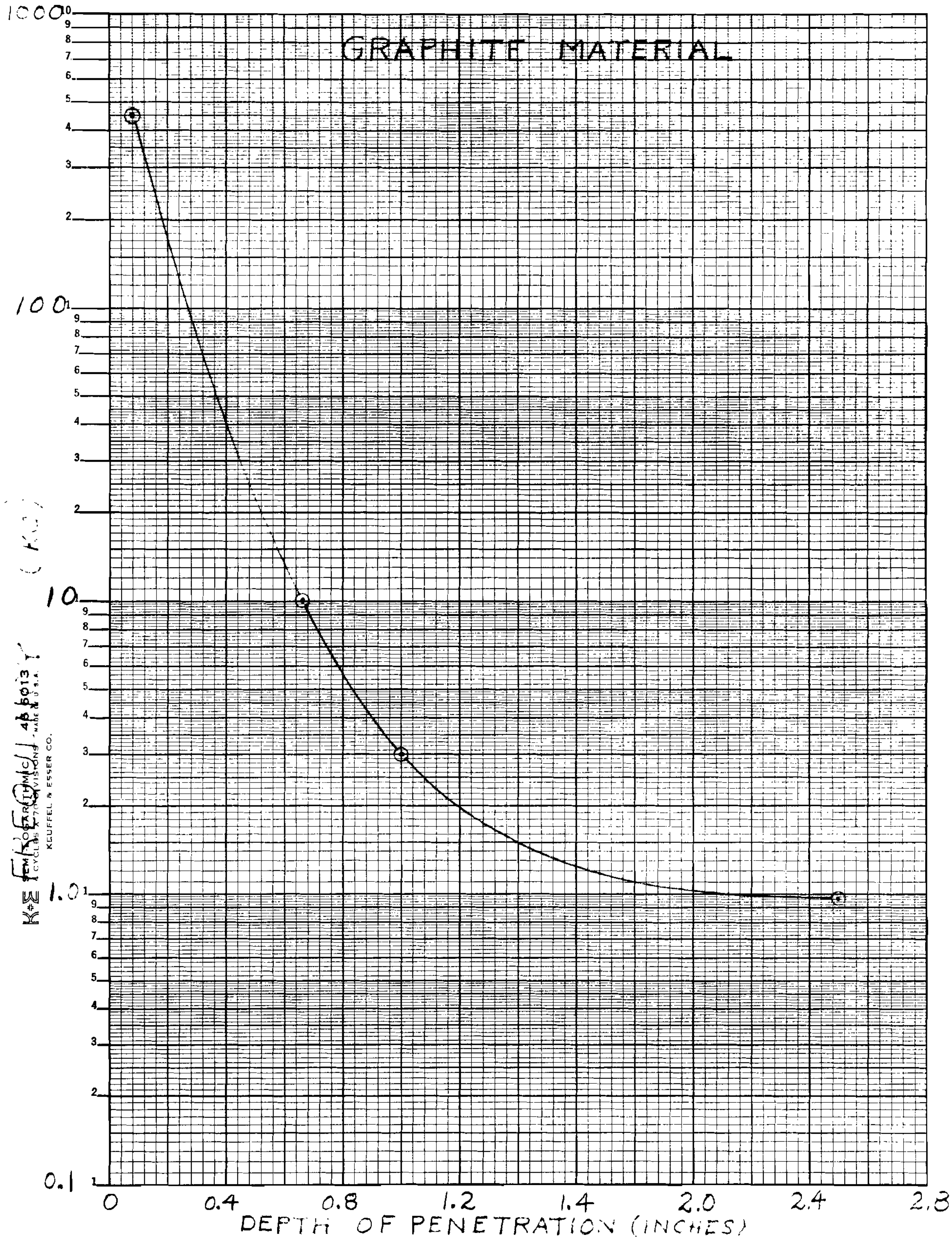


Fig. 15. Penetration depth of rf field in graphite as a function of frequency

In addition, the coupling between the work coil and the susceptor is also affected by the frequency used. The coupling, which is the distance from the work coil inside diameter to the susceptor outside diameter, would have to be closer with the higher frequency to be efficient. If the work coil is close to the susceptor, less thermal insulation can be used on the outside diameter of the susceptor, resulting in more heat loss and thus much greater capacity in the power source to overcome the losses. For these reasons, then, the lower frequency unit was chosen for this part of the program.

The motor generator set was chosen over a solid state unit because it is considered more reliable. The solid state unit is prone to failure of various components, notably the SCR's and diodes. It is necessary to select a unit that will run continuously without interruption or repairs.

The 175 KW, 960 cycle, motor generator set has an excellent depth of penetration into the graphite susceptor and has also good stability against input power fluctuations. This power source has a large coupling distance and permits use of up to 2.5 in. of insulation on the outside of the susceptor.

The vendor selected to construct the 175 KW, 960 cycle power source, which included (1) a Westinghouse horizontal water cooled motor generator set, (2) a control cubicle which houses the gauges, transformer, and capacitor bank, (3) a power feedthrough, (4) a reduced voltage manual starter, and (5) a 26-in. I. D. electrically insulated copper work coil, was Ecco High Frequency Company, North Bergen, New Jersey. The original copper work coil purchased was 26 in. I. D. by 36 in. long and was later replaced by a work coil 26 in. in diameter by 12 in. long. The long coil raised the temperature too high in the afterheater section causing melting of the seed while melting the sapphire charge in the crucible. The shorter coil heats mainly the susceptor around the crucible, while the afterheater section is heated mostly by conduction and radiation.

The control crucible is equipped with an automatic Leeds & Northrup controller using an Ircon as the sensing head. Some problems were encountered in use because the windows of the chamber became clouded during operation, thereby reducing the measured temperature and causing the temperature to increase in the crucible area. The power supply has been operated manually very efficiently, and when the conditions become stabilized, no further changes are required to hold a constant temperature.

Figs. 16 and 17 are photographs of the various components that are included in the power source: (16) reduced voltage manual starter and 175 KW, 960 cycles Westinghouse horizontal water cooled motor generator set, and (17) the Ecco High Frequency control cubicle which houses the capacitors, variable transformers, and various meters. The above-described heating unit has heated the large 12-in. diameter crucible and components with a charge of 10,000 g of sapphire boules in excess of 2050°C in one hour. With some slight design change in the short 12-in. coil, the time could be decreased if necessary.

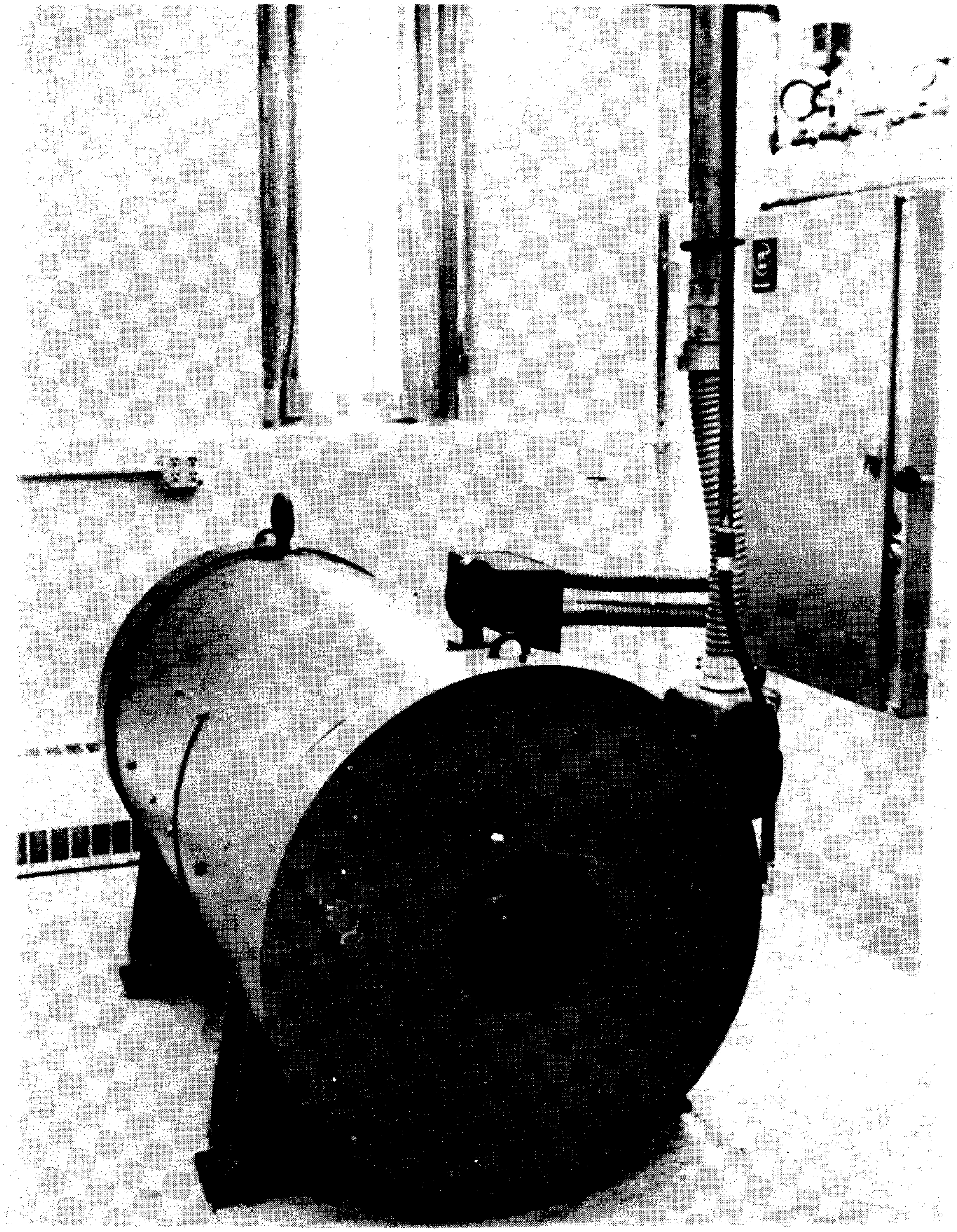


Fig. 16. 175 KW, 960 cycle Westinghouse horizontal water cooled motor generator (the reduced voltage manual starter is visible on the wall at right

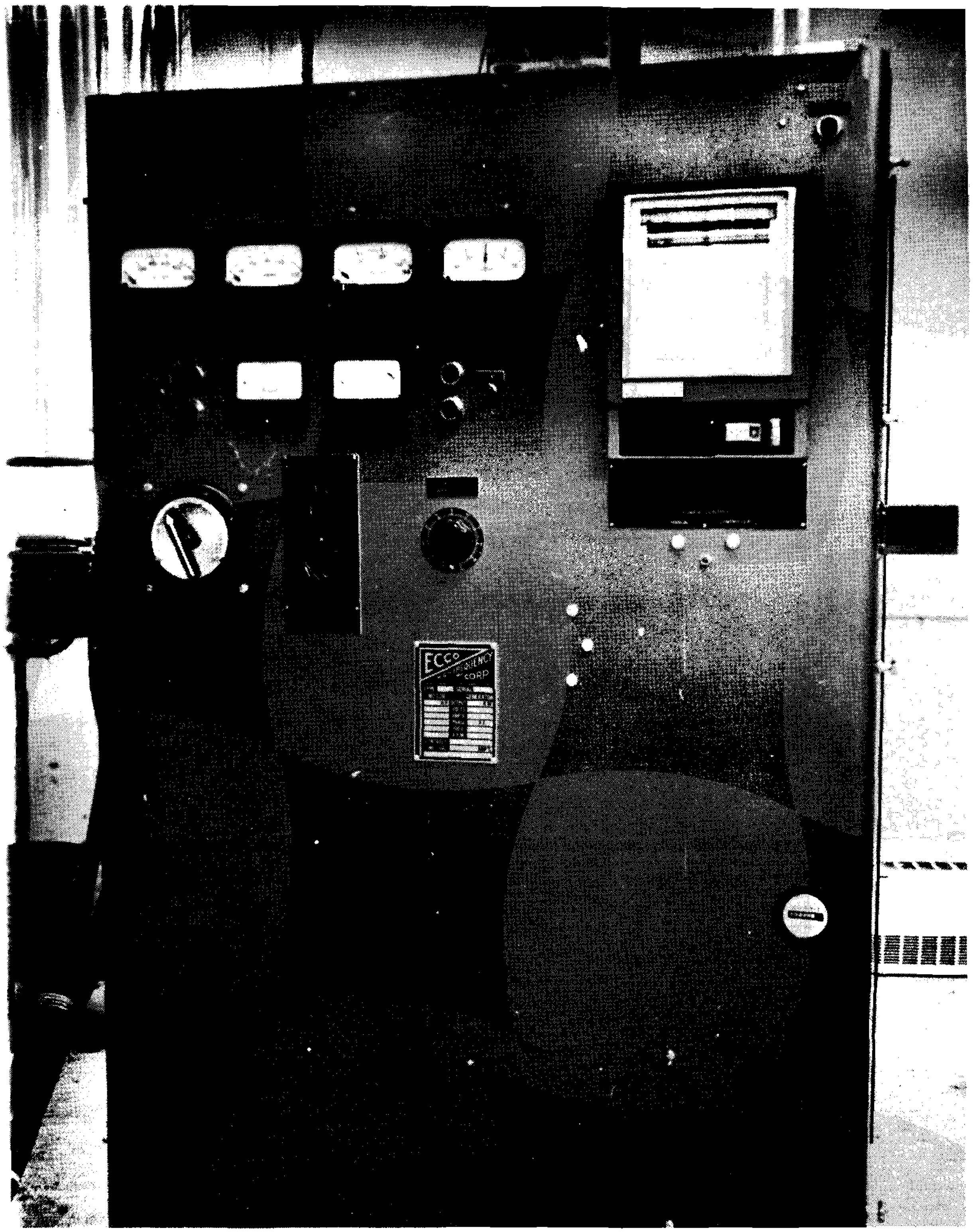


Fig. 17. Ecco high frequency control cubicle

2. Furnace enclosure

The second major change of the standard apparatus adopted for growth of large plates was the type and configuration of the furnace chamber. The standard apparatus for growth of much smaller configurations has the rf coil externally wound about a double-walled, water-cooled quartz chamber. In the present case, it was concluded that any similar design would be unwieldy and totally impractical. Consequently, the design approach was to build a water-cooled stainless steel enclosure with the work coil on the inside. Among the major advantages, this approach allows all of the space between the work coil and the work to be occupied by insulation, thus reducing greatly the power input required.

The large furnace chamber, shown in Fig. 18, consists of an inner stainless steel chamber, 52 in. diameter by 58-1/2 in. high. The outer jacket is constructed of carbon steel, with channeling between to direct the flow of water for efficient cooling. The water comes in at the bottom and exits at the top of the chamber. The enclosure has three sight ports, two being 4 in. in diameter and one 7 in. in diameter, with stainless steel shutters for thermal protection. The large sight port is located in the front of the unit to enable viewing of the sapphire plate during the growth cycle.

The bottom of the furnace enclosure is mounted on a hydraulic scissor lift, electrically operated, 3000-lb capacity, to raise or lower the bottom dished head carrying the contents of the furnace. The bottom dished head is held in position by 6 clamps mounted on the flange welded to the main chamber. These clamps hold the bottom dished head in place when back filling with argon and during the growth cycle. The 26-in. I. D. coil is mounted on a stainless steel, water cooled pedestal plate.

Fig. 19 illustrates the setup of the graphite and metal components inside the insulated copper work coil. Several layers of graphite felt, item 16, are placed on the surface of the water-cooled pedestal plate. The bird cage support structure, item 3, using grade AUC graphite, rests on the above graphite felt. There are four layers of 1/4 in. thick graphite felt around the outside of the birdcage structure. On the inside of this structure are five pillars constructed of graphite felt rolled together to support four layers of graphite felt, as shown in Fig. 19, item 16. This setup, using the 1/4 in. thick graphite felt, requires replacement every three months of operation to insure the proper insulation values required.

The graphite susceptor, item 5, using purified graphite from Airco Speer, grade 873 RL, rests on the birdcage structure and is wrapped with eight layers of 1/4 in. thick graphite felt insulation. A shelf is machined on the inside of the graphite susceptor to locate and support item 6, the graphite pedestal block machined from Airco Speer, grade 873 RL, purified material. Recesses are machined into the graphite pedestal block to locate the crucible support rods, item 15, permitting the atmosphere to circulate around the crucible for a more uniform temperature. Small tungsten pads, item 7, protect the tungsten crucible from coming into direct contact with the graphite.

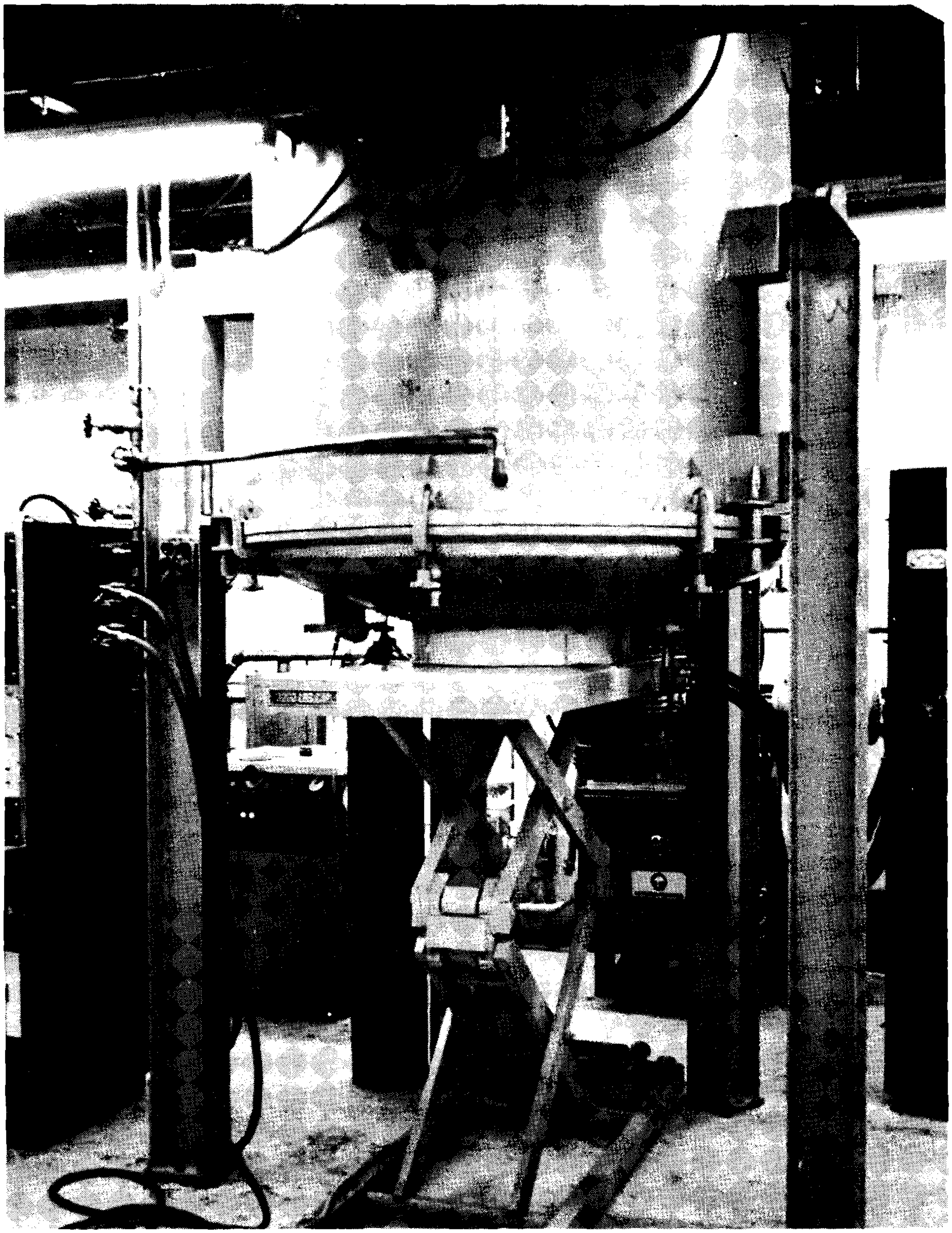


Fig. 18. Furnace chamber (this photograph shows the bottom dished head raised and clamped into position, still on the hydraulic scissors lift)

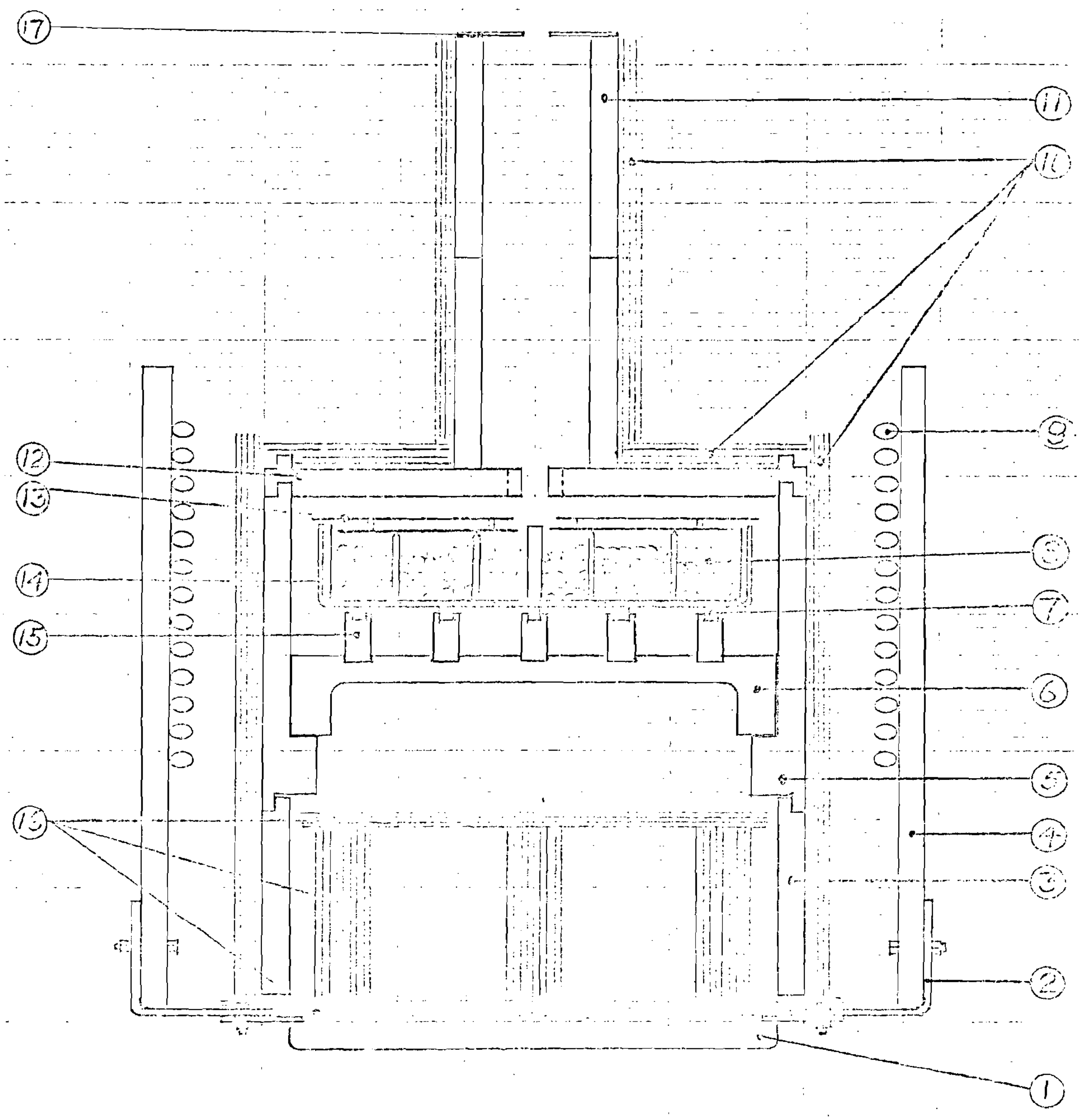


Fig. 19. Setup for full-scale plate growth

Fig. 19. Description

Item No.	
1	Stainless steel water-cooled pedestal plate
2	Stainless steel coil mounting brackets
3	Bird cage support structure - grade AUC graphite
4	Transite work coil supports
5	Graphite susceptor - grade 873 RL
6	Graphite pedestal block - grade 873 RL
7	Tungsten support pads
8	Sapphire charge
9	Insulated copper work coil
10	Graphite felt insulation
11	Graphite afterheater sections - grade 873 RL
12	Graphite susceptor cover - grade 873 RL
13	Tungsten cover and shield assembly
14	Tungsten crucible
15	Graphite support rods - grade AUC
16	Graphite felt insulation
17	Graphite top covers

The tungsten crucible containing a molybdenum liner, an orifice, cover supports, and covers rests on these support rods equally spaced on the inside of the graphite susceptor. A graphite cover, item 12, is located on the graphite susceptor by means of a tongue and groove arrangement. The two graphite afterheater sections, 8 in. long, rest on the cover, with the inside equally spaced from the orifice. Three layers of 1/4-in. thick graphite felt insulation are placed on top of the graphite cover and three layers are wrapped around the afterheater sections.

A 6-in. diameter flange on the top of the furnace enclosure locates and secures the crystal puller. Fig. 20 is a photograph of the finished crystal puller that is mounted on the top of the enclosure. The crystal puller has a speed variable from 0 to 8 in./hr using a Bodine constant torque motor with a Minarik controller. A shaft extends down from the bottom of the crystal puller to where the seed holder assembly, Fig. 21 is attached. This seed holder incorporates a mechanism which prevents orifice damage when the seed is lowered against it. In this operation, the setup is heated until the top of the orifice reaches the melting temperature of sapphire. The seed is then lowered into contact with the orifice and is, in fact, held against it to melt back the seed sufficiently to insure complete contact of the seed with the orifice and to provide additional liquid to connect the seed with the meniscus and orifice capillaries. Danger to the orifice is greatest at the moment of initial contact and is the result of the extreme plasticity of molybdenum at that temperature compared to the strength and hardness of the relatively cold sapphire seed crystal. The feature which prevents this type of damage is a vertical slip arrangement in the seed holder which prevents more than the actual weight of the seed from pushing down on the orifice. The seed holder accepts flat (in the horizontal plane) seeds, 0.300 in. thick, which are attached by two 1/8 in. diameter sapphire pins.

The furnace enclosure contains a power feedthrough and a multipin feedthrough in the bottom dished head. A thermistor gauge tube and penning gauge tube are located in the walls near the top of the furnace enclosure. There are a number of other openings in the chamber for gas inlet, pressure gauges, instrumentation feedthrough, vacuum gauge, and the vacuum system.

The furnace enclosure is supported on four legs, sufficiently spaced to permit lowering the bottom dished head and movement from beneath the main enclosure for removal of internal components for cleaning or replacement.

3. Vacuum system

The furnace chamber has a 10-in. diameter opening for connection to the vacuum system. The vacuum system consists of a Leybold-Heraeus 88 CFM mechanical pump connected to a 685 CFM mechanical blower, as shown in Fig. 22. The pumping system leads directly to the furnace enclosure through a 4-in. electrically operated pneumatic vacuum valve and a 10-in. electrically operated pneumatic angle valve, as shown in Fig. 23. A 4-in. diameter copper line connects the various components with proper flanges and O-ring seals. The 10-in. electrically operated penumatic vacuum angle valve was manufactured by NRC, Norton Company, Vacuum Equipment Division.

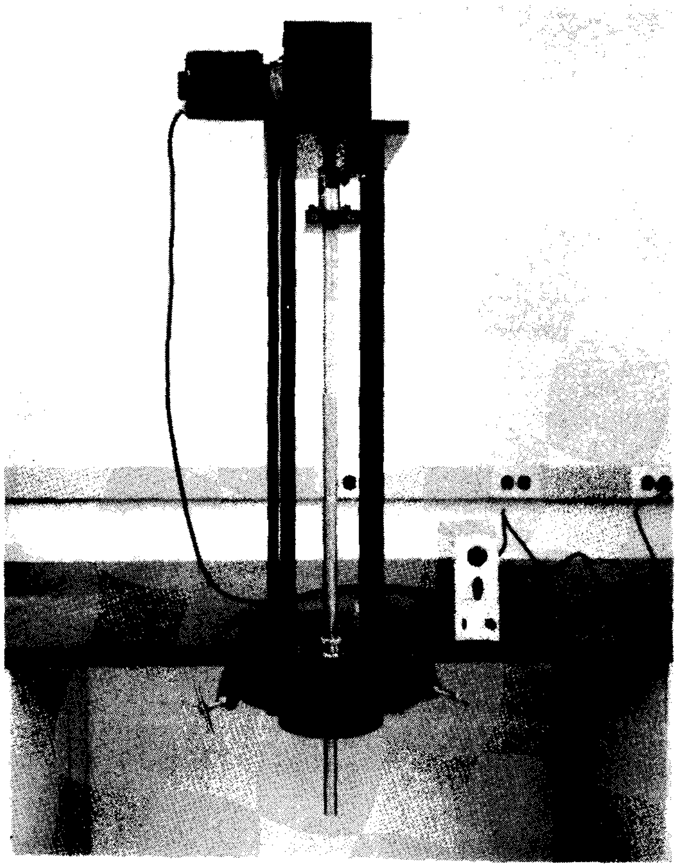


Fig. 20. Crystal puller

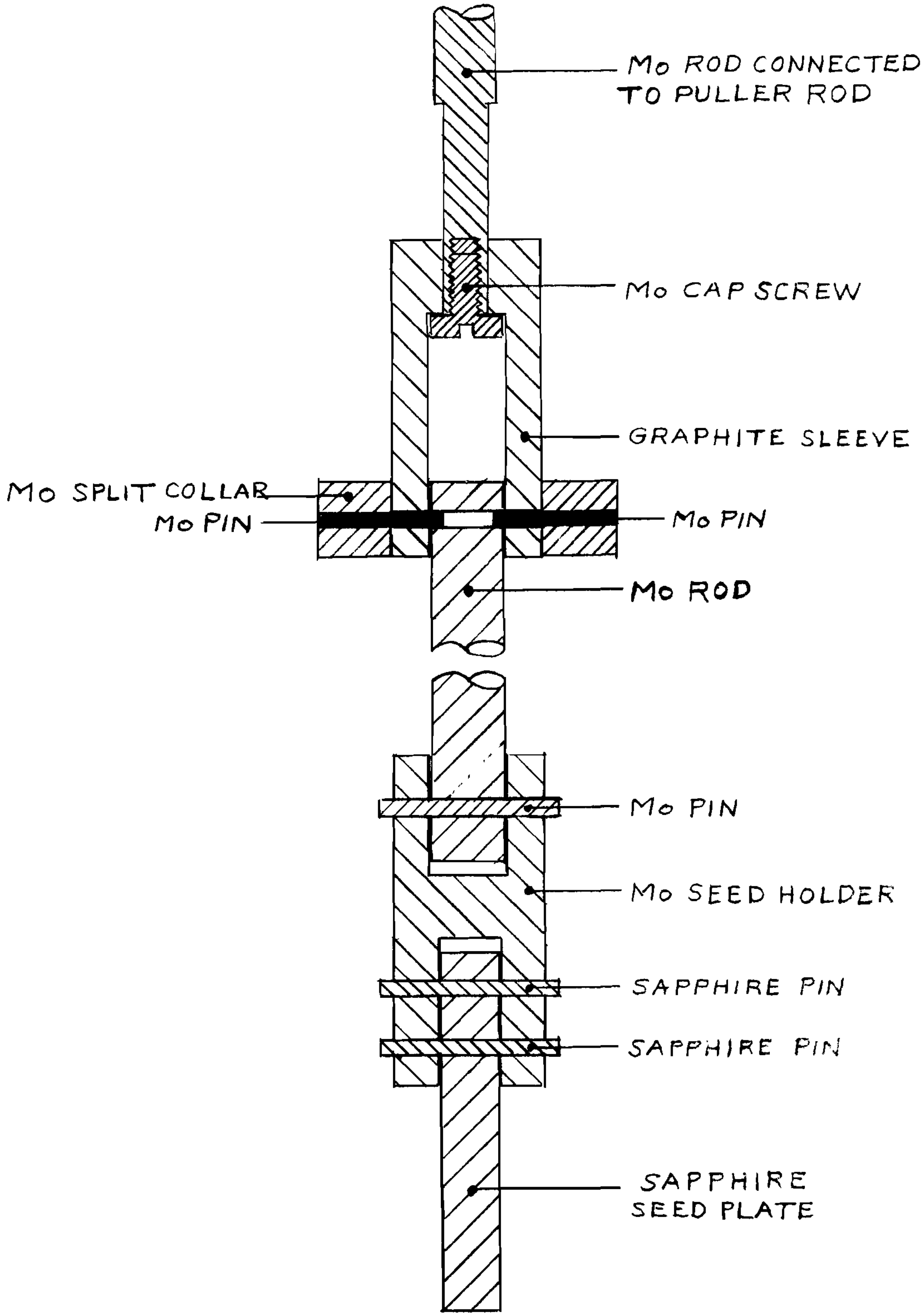


Fig. 21. Seed holder (the usual form of the seed is a plate, as illustrated here end-on)

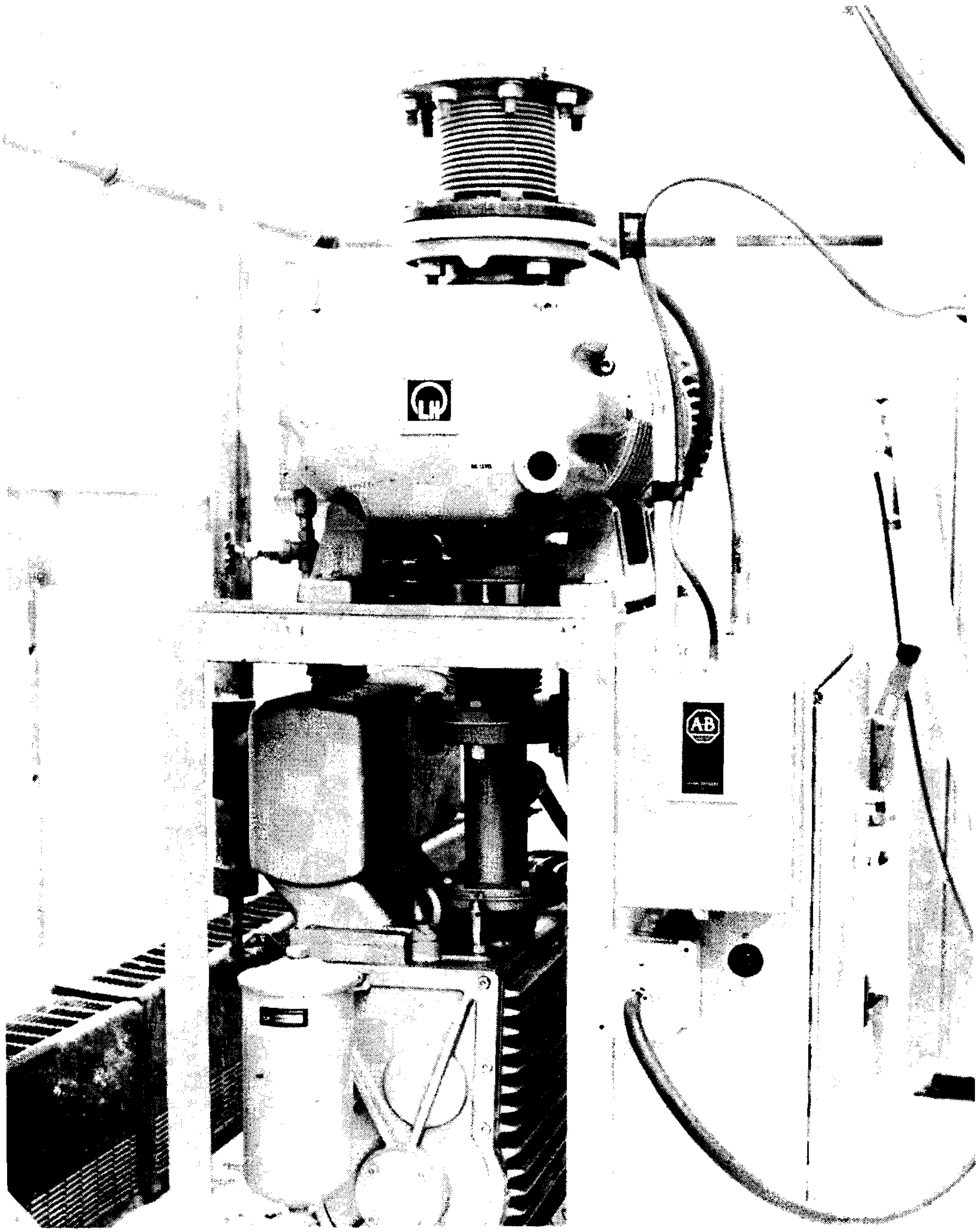


Fig. 22. Mechanical pumping system. The mechanical pump is at the bottom, and the blower, at the top

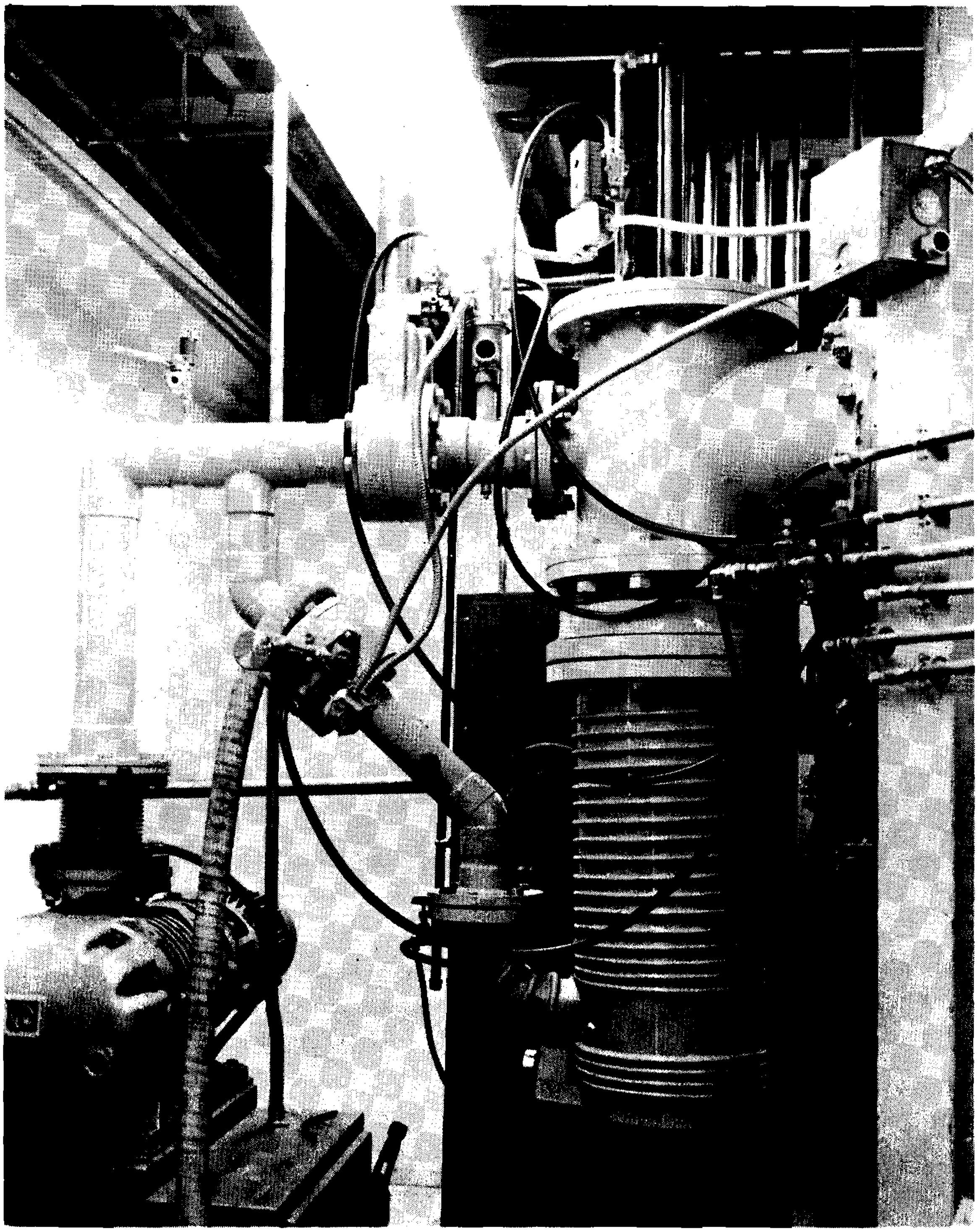


Fig. 23. Vacuum diffusion pump and associated valving

Sealed to the bottom flange of the angle valve is a Leybold-Heraeus water-cooled baffle and a 10-in. CVC diffusion pump manufactured by Bendix Scientific Instrument and Equipment Division, Fig. 23. The diffusion pump is equipped with a fluid level gauge and a thermostat that will shut off the power to the pump heaters if sufficient cooling water is not available to the pump cooling coils. A roughing line having a 3-in. electrically operated pneumatic vacuum valve is inserted between the roughing side of the diffusion pump and the vacuum pump side of the 4-in. vacuum valve, Fig. 23. This permits the pumping path to bypass the diffusion pump at an intermediate vacuum or to go through the operating diffusion pump when higher vacuum is required. A 5.6 CFM Welch vacuum pump is connected to the 3-in. diameter line between the 3-in. vacuum valve and the diffusion pump to permit the isolation of the diffusion pump if the chamber required opening. This small vacuum pump is commonly called a hold down pump.

The electrically operated pneumatic vacuum valves are operated manually or automatically from the control panel cabinet (Fig. 24) which houses the vacuum gauges and water monitoring temperature gauge. The thermistor gauge is housed in the panel control cabinet and will indicate the vacuum down to 1 millitorr, while the Penning gauge will indicate the vacuum down to 1×10^{-7} torr. These gauges were purchased from Bendix Scientific Instrument and Equipment Division.

The vacuum furnace comprising the furnace enclosure pumping system, and rf supply, has proven completely satisfactory and relaible. Indeed, since the initial de-bugging period, no breakdowns or delays caused by malfunctions of this apparatus have been experienced.

4. Crucible fabrication

Both molybdenum and tungsten are satisfactory container materials for molten alumina in terms of their compatibility with the liquid. However, other considerations must be invoked since the actual choice is quite important. Molybdenum is always the choice, when possible, because it is cheaper and more readily machined. Molybdenum is always used in small setups for those reasons.

The primary reason for which tungsten is sometimes used is related to the method of heating and the geometry of the setup. The production of heat is almost entirely in the walls of the susceptor around the periphery of the crucible. Heat flow is therefore inwards toward the center of the crucible from its walls. Heat is lost from the setup both at the top and at the bottom. The result is, by definition, a temperature gradient from the susceptor towards the crucible. The larger mass of the crucible and charge results in a greater thermal gradient during heating. The larger area for heat to be lost above and below the crucible similarly results in a greater thermal gradient during steady-state operation. Since the end temperature required for growth is always the same, the walls of the susceptor are always hotter the larger the crucible which is being heated.

Molybdenum and carbon undergo an eutectic reaction at approximately 2200°C. This temperature is probably always exceeded, even in small setups; but by physically separating the two components, it can be suppressed in the small setups. However, even in the 2-to -3 in.

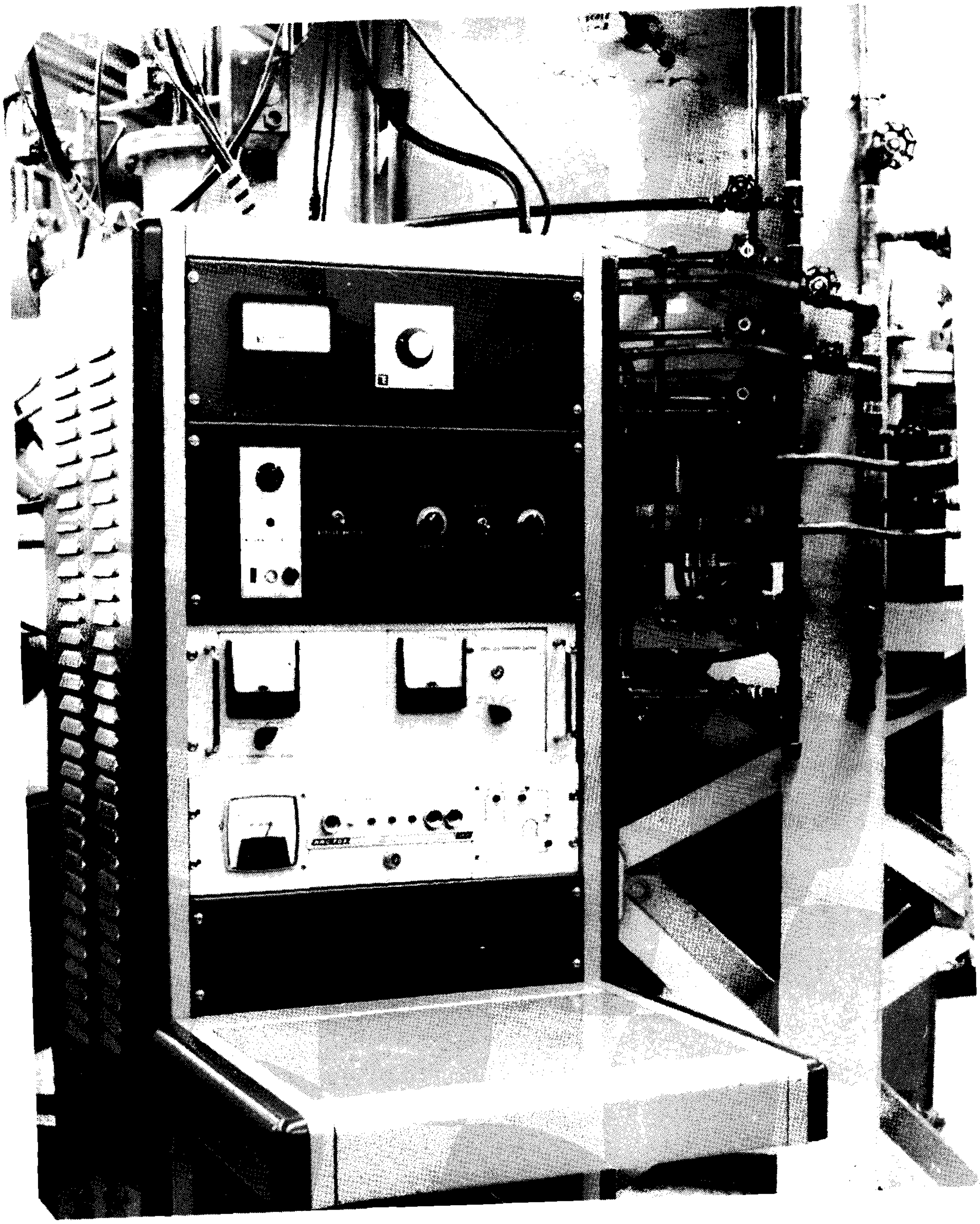


Fig. 24. Panel control cabinet. These controls are for the vacuum system and also for the crystal puller.

plate setups, it was found necesary to use a tungsten crucible (which undergoes a similar reaction, but at nearly 2500°C) because of the higher temperature. Thus tungsten was adopted in the present case and has proven satisfactory.

The initial approach to the fabrication of a crucible was a weldmet structure. In fabricating rectangular-shaped crucibles, sheet tungsten was cut by one of two methods: hot shearing or a rotary carborundum saw blade. The hot shearing method caused delaminations within the sheet tungsten, especially if the gap between the shear blade and the table of the shear exceeded approximately 0.003 in. The cutting with a rotary carborundum saw blade was more costly because of the increased time involved, but every piece was acceptable. The crucibles were electron beam welded, and because of their (relatively) small size, as shown in Fig. 25, the welded edges were satisfactory. This crucible was 3-1/2 in. long × 1 in. wide × 2 in. high to grow plates up to 3 in. wide × 3/8 in. thick.

The scale-up of this rectangular-shaped crucible using electron beam welding as the fabrication method presented a number of problems. Due to the greater distance in the weld line, an increased number of stresses were set up in the tungsten sheet. This led to cracks in or near the weld area after one or two growth cycles. One of the 6 in. long × 1 in. wide × 2 in. high weldmet tungsten crucibles lasted 8 runs before failure along the electron beam weld at the bottom. The difference in the coefficient of thermal expansion between tungsten and sapphire helped to propagate the cracks once initiated until the crucible would not contain the molten sapphire material.

Another method of welding, TIG welding, reduced the stresses set up in the first welding process because the TIG process heats a larger value of material than the electron beam method. A crucible 7 in. long × 3 in. wide × 3 in. high was successfully fabricated by TIG welding. The follow on attempts to fabricate a 7 in. long × 3 in. wide × 3 in. high tungsten crucible by TIG welding ended in a catastrophic failure, and therefore this method was abandoned.

Other methods of fabricating tungsten crucibles, such as spinning and hot forming, were then investigated. These methods required a shape change as they are possible only for a circular configuration. A weldmet circular tungsten crucible, 3-1/2 in. in diameter, was tried using electron beam welding, resulting in many cracks throughout the bottom area and some traveling up the sides. A second 3-1/2 in. diameter circular tungsten crucible was fabricated after stress relieving all parts before electron beam welding, and is shown in Fig. 26. The tungsten crucible had no visible crack; but when it was heated for the first time, cracks developed.

Subsequently, all attempts to fabricate crucibles by any process involving welding were abandoned. Two sources for spun crucibles were considered and crucibles were obtained from both of them. Newark Spinning & Stamping Company successfully spun two 7-in. diameter, 3-in. high, tungsten crucibles early in the program but was unable to do so successfully at a later time. Metallwerk Plansee in Austria was able to fabricate crucibles of both this size and larger ones having a 15-in. diameter on a reliable basis. This vendor is the only one known at this time capable of reliably supplying formed circular tungsten crucibles.

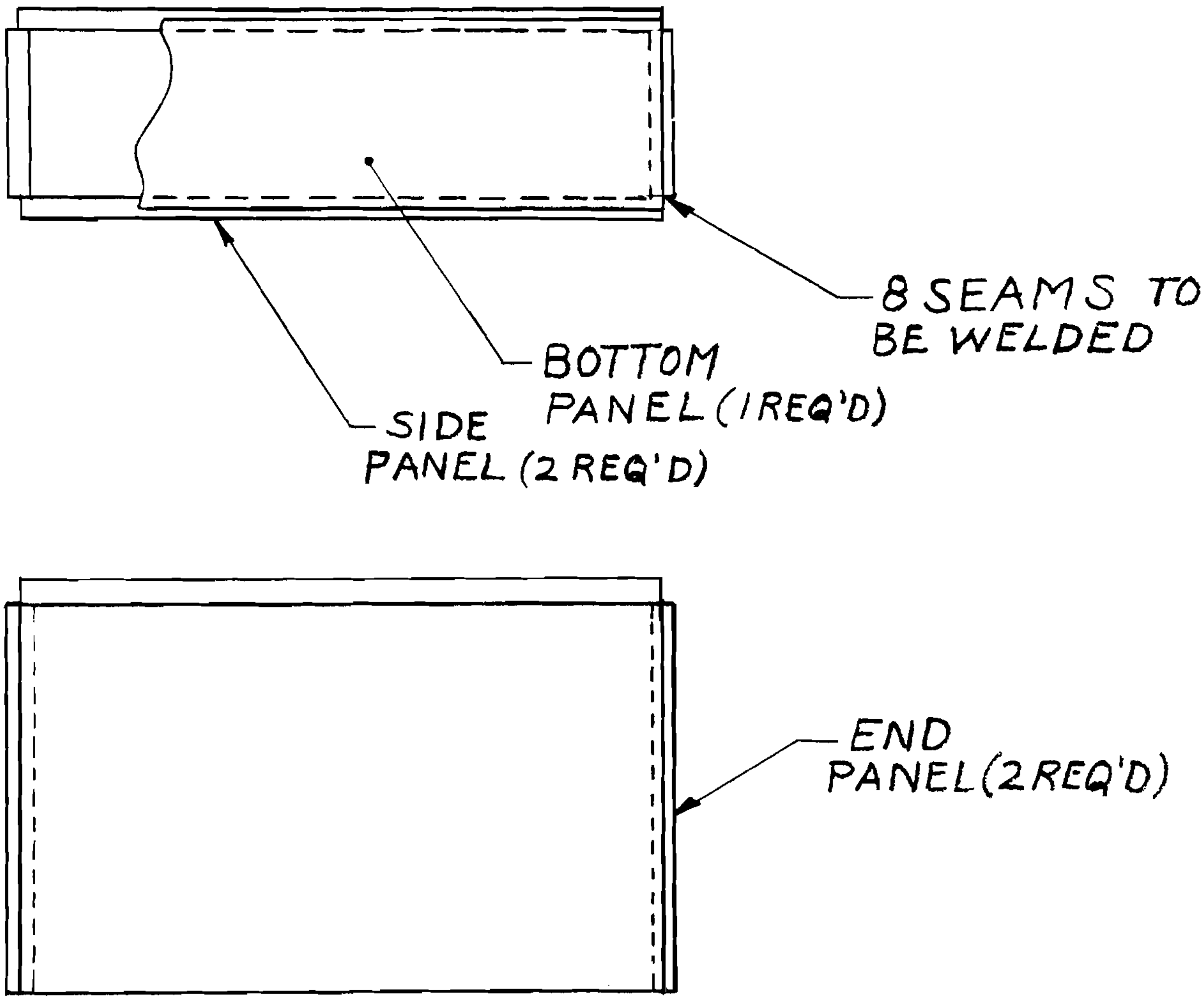


Fig. 25. New crucible design showing overlapping seam construction for electron beam welding

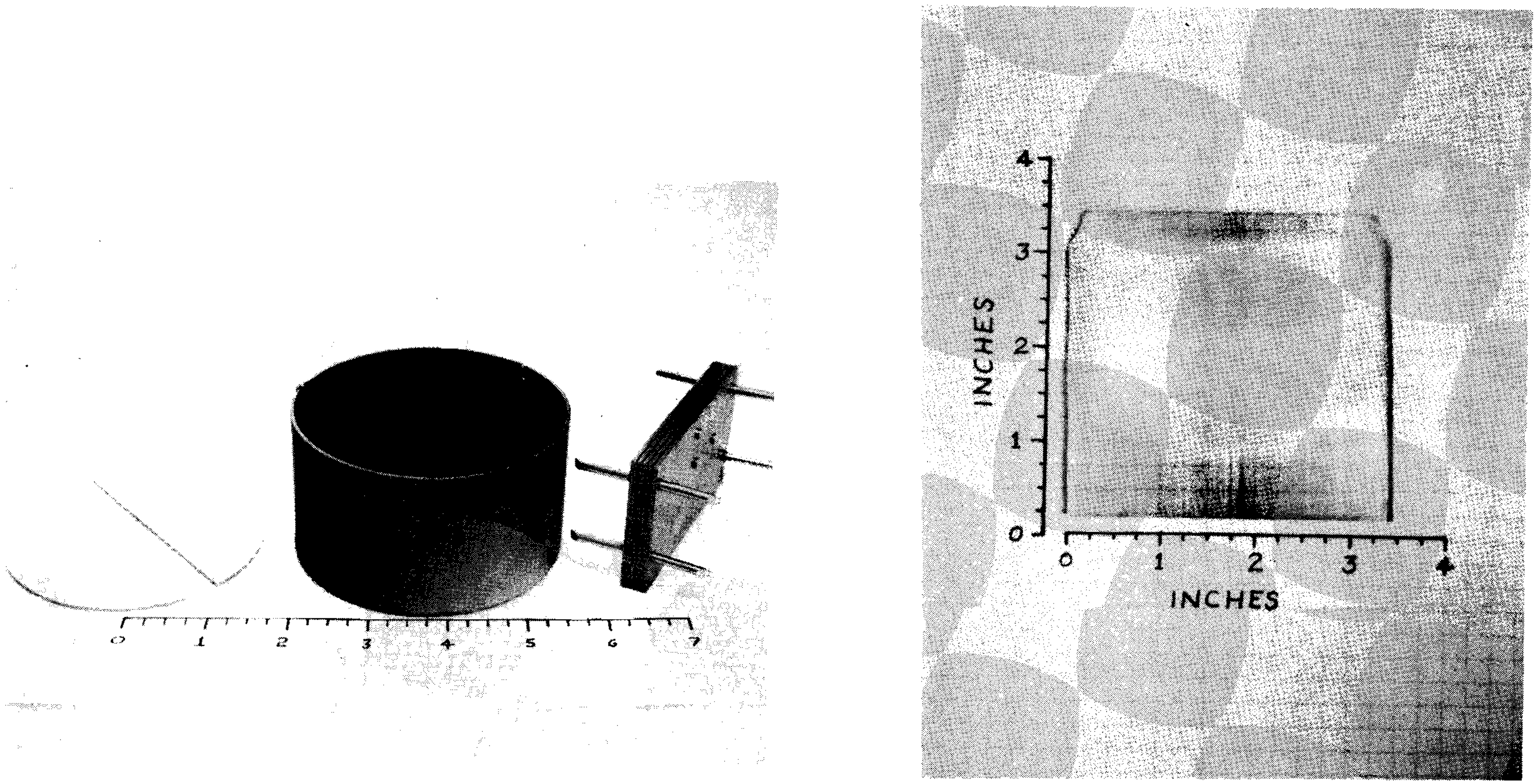


Fig. 26. Experimental round electron beam-welded tungsten crucibles and associated components

The 7-in. diameter tungsten crucibles had wall thicknesses of 0.040 in. which deformed during continuous use, and this deformation had an effect on the thermal stability in the orifice area. The large 15-in. diameter tungsten crucibles had wall thicknesses of 0.125 in. which did not deform over the course of several heat cycles. It is apparent that it is a firm requirement to have heavy wall tungsten crucibles to eliminate wall deformation for continuous heat cycles. The one large crucible has had 6 complete growth cycles without any noticeable deterioration. This crucible is illustrated in Fig. 27.

Adoption of the circular geometry had a side benefit which resulted from its increased charge volume capacity. The rectangular crucibles which we had previously considered had a limited charge capacity and would have required replenishment during the growth run. However, the capacity of the circular spun crucibles was great enough to contain initially even more charge than is required to grow the largest plate. Thus, the need for continuous feeding of the molten alumina during the run was obviated.

The use of molybdenum liners in either molybdenum or tungsten crucibles is sometimes adopted for various sapphire growth setups. Liners are spun, have much thinner walls than the crucibles, and, as a result, can be replaced at relatively low cost compared to the crucible. In this respect, tungsten crucibles prove to be far more durable than molybdenum ones, since one of the two usual sources of crucible damage is caused by overheating and subsequent melting or reaction with the susceptor. The second source of crucible damage results from loss of the orifice when a substantial charge remains in the crucible, making the whole setup scrap if no liner is used

We therefore adopted the use of liners as part of the plate growth process. These liners had 0.040 in. wall thickness and were obtained from Newark Spinning & Stamping Company. As a result of the use of liners, none of the tungsten crucibles of the final design were lost to other than normal wear and tear.

5. Orifice fabrication

During the design and fabrication of the large crystal-pulling machine, many plate growth runs were carried out in the small crystal growing machine which used the 50 KW, 450 KC rf generator. These experiments were carried out to investigate the effects of orifice design, orifice materials, and, to a limited extent, susceptor and afterheater configurations on the growth characteristics of sapphire plates.

Molybdenum and tungsten were compared as orifice materials. In the growth of 1-1/2 in. wide sapphire plates, a molybdenum orifice produced, with no difficulty, a crack-free sapphire plate 4 in. long. A tungsten orifice produced considerable difficulty in attempts to grow a crack-free sapphire plate. Tungsten has a higher thermal conductivity than molybdenum so that it must be hypothesized that in some way this difference was responsible for establishing a greater thermal gradient when tungsten was used as an orifice. As a result of these tests, molybdenum was adopted as the orifice material for the larger-sized sapphire plates.

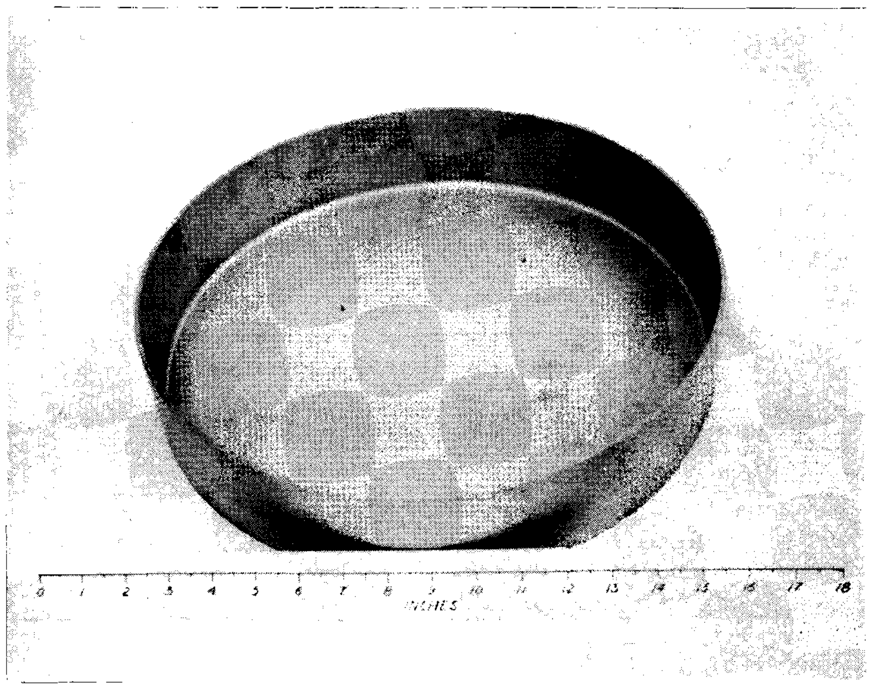


Fig. 27. Full-size tungsten crucible fabricated by spinning

A new orifice design was evaluated by growing a plate 2-1/4 to 2-1/2 in. wide × greater than 4 in. long from our standard size tungsten crucible 3-3/8 in. long × 13/16 in. wide × 2 in. high. The first attempt was an orifice constructed with molybedenum plates 3 in. long × 2 in. wide, separated by 1/4-in. wide × 0.040 in. thick spacers on the ends. There was an unsupported distance of 2-1/2 in. between these spacers. The first attempt to grow a sapphire plate resulted in distortion of the orifice plates, varying the thickness of the grown sapphire plate from the end to the center. The second orifice in this configuration was constructed from five 0.050-in. thick molybdenum plates, 3 in. long × 2 in. high, and four 0.040-in. thick molybdenum spacers on each end. This second orifice also contained short tubular molybdenum spacers (1/4 in. O. D. × 1/8 in. I. D. × 0.040 in. long) at several intermediate locations between the plates: three spacers between each outer pair of plates (numbers 1 and 2, 4 and 5) and one spacer between each inner pair (numbers 2 and 3, 3 and 4). The second orifice produced a satisfactory sapphire plate 2-1/4 in. wide × 4 in. long × 0.410 in. thick, without distortion of the molybdenum orifice plates.

It was necessary to evaluate the thickness of the feed capillary to insure the proper rise of the molten sapphire in the intermediate and full-scale orifice designs. The thinner the feed capillary, the higher the liquid will rise. Calculations indicated the width of the capillary would be 0.015 in. to 0.020 in. The balance of the tests run on the 3-in. wide plates incorporated a 0.020-in. wide capillary.

The molybdenum spacers used in the central region of the orifice were found to be associated with vertical gray streaks in the grown plate. This problem was obviated by using tungsten for the same function. However, it is obvious that the success of this change cannot be ascribed to chemical differences between tungsten and molybdenum. It is unlikely that thermal differences are responsible either for the improvement. At the present time it seems more likely that surface impurities in the molybdenum, possibly the result of the machining operation, were responsible for the discoloration.

Orifices having one to four capillary slots were fabricated and used to grow plates in order to investigate the effect of the capillaries on the growth. Results of these experiments in both the laboratory and the large plate growth apparatus showed that thermal gradients along the long direction of the orifice were reduced in proportion to the number of capillary slots used. Thus, the majority of the 6-in. and 12-in. orifices used were constructed with 4 capillary slots, as shown in Fig. 28, for a 3-in. orifice.

With the above spacing defined except for the width of the capillary which became 0.015 in., the intermediate size orifice, 6 in. long, and the full-size orifice, 12 in. long, were designed using the same parameters. Fig. 29 illustrates the full-size molybdenum orifice designed with 0.015 in. wide capillary and spacers between the plates to eliminate deformation within the fabricated structure. The orifice design appears to require additional design work to eliminate the streamers of small voids scattered throughout the plate which reduce the clarity. Large sapphire plates have been grown to more than 12 in. wide × 0.440 in. thick, as shown in Fig. 30.

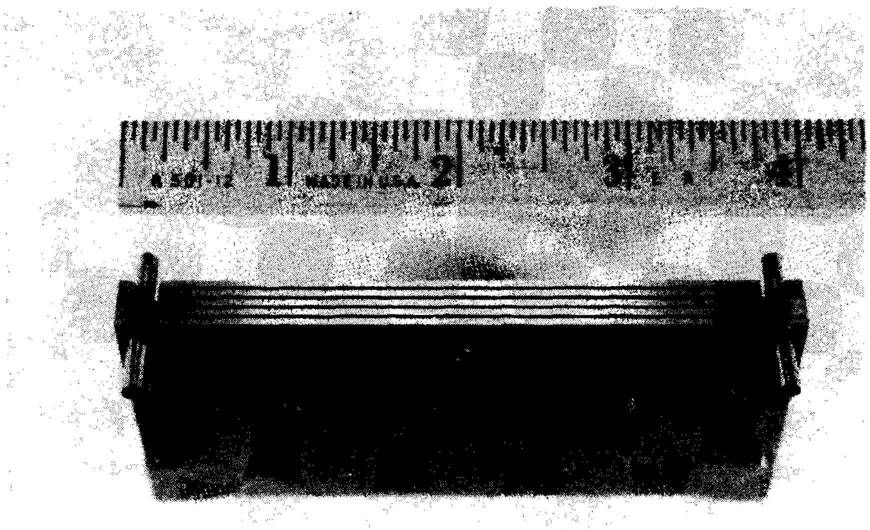


Fig. 28. Typical test orifice design

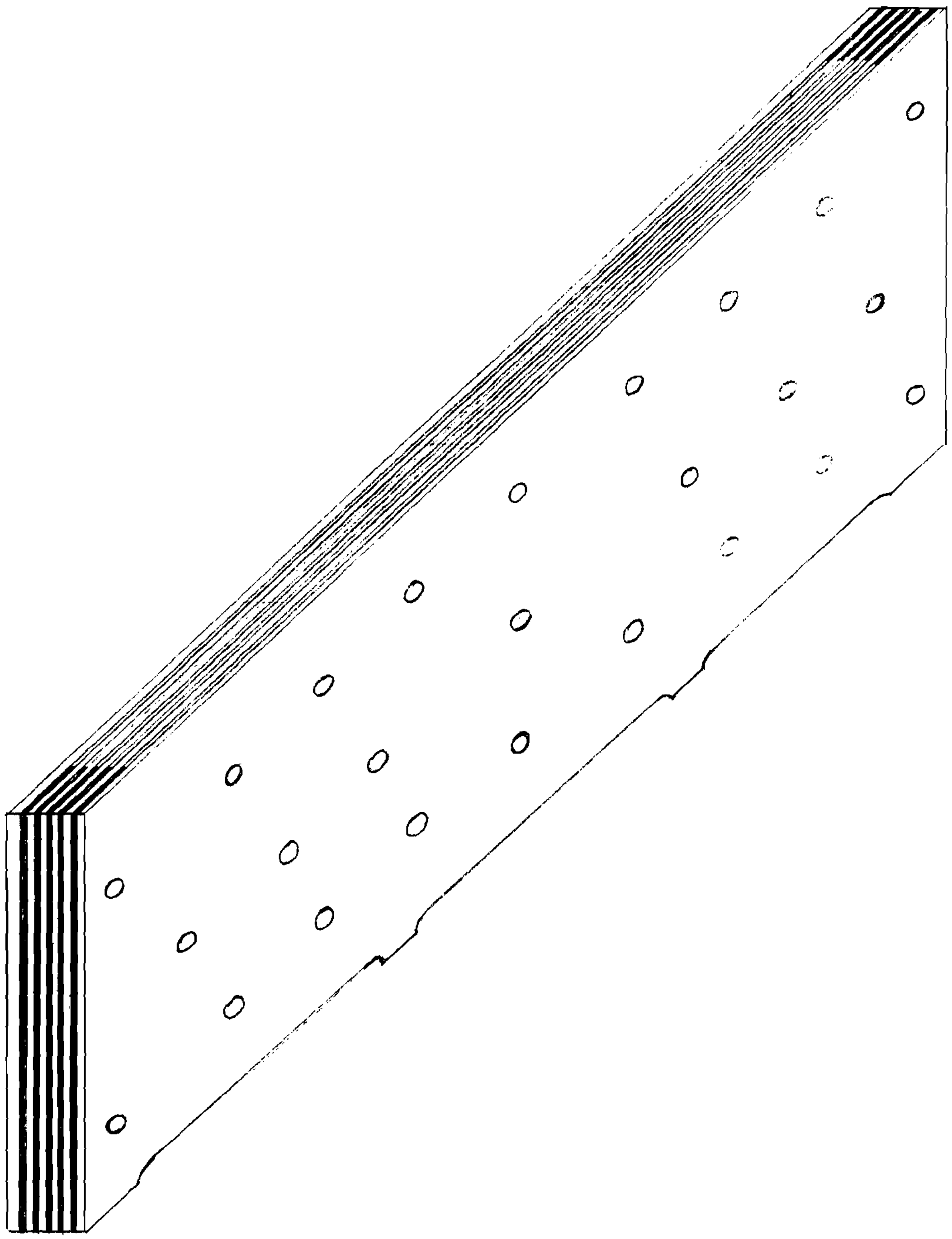


Fig. 29. Full-scale orifice for sapphire plate growth

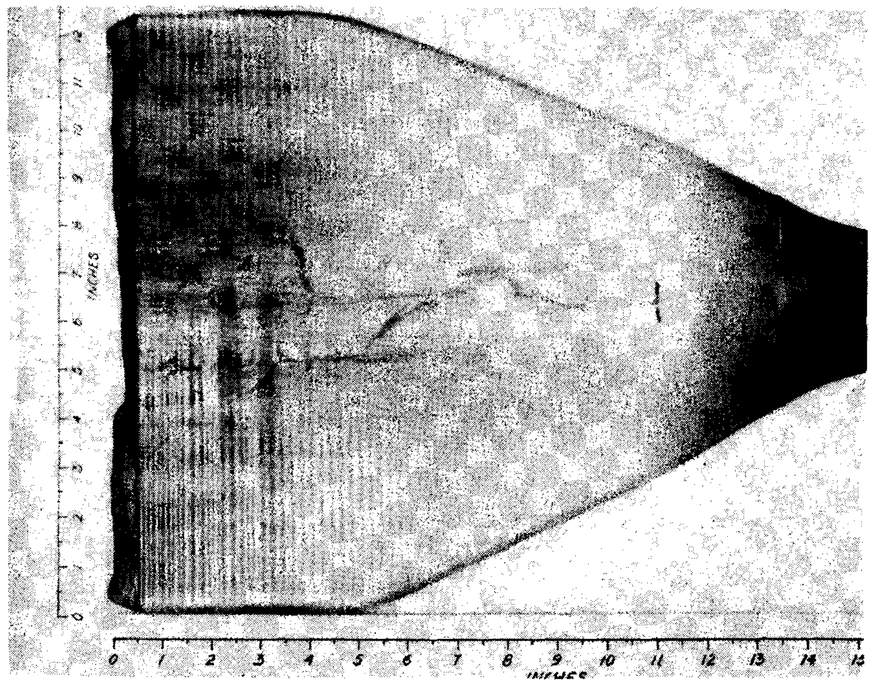


Fig. 30. The largest sapphire plate grown in the full-scale setup

6. Heat shielding

Heat shielding of two types is used to maintain the proper vertical temperature gradient to allow growth to proceed and to maintain a level horizontal temperature distribution to prevent cracking. Horizontal tungsten heat shields are used for the first of these functions. Here tungsten is used rather than molybdenum because it is stronger at temperature and thereby helps to resist deformation. Metal is required for these shields as they are placed immediately above the charge in the crucible. Vertical graphite heat shields are used below the afterheater and above the tungsten shields to control the horizontal temperature distribution. Here, graphite has proven a very satisfactory shield material and undergoes no distortion in use.

The metal shields are comprised of two tungsten sheets, one above the other, and are made in sections both for ease of manufacture and for simplicity in installation. Fig. 31 shows a welded assembly of tungsten support pins which is immersed at its lower end in the molten alumina and supports the shield assembly above the charge. The shield assembly is shown in Fig. 32. Both of these assemblies are illustrated for the full-size crucible. The subscale crucible utilized a similar arrangement of metal shields. These tungsten shield assemblies appear to be satisfactory for plate growth.

The graphite shields provide the main mechanisms for the alteration of the temperature distribution. Since, without insertion of these shields, the hot susceptor walls directly face the growing plate between the orifice and the bottom of the afterheater, it is possible to add heat to this area by manipulation of these shields. Fig. 33 illustrates the configuration of the graphite shields used in the subscale growth configuration. In their use, these shields were attached to the graphite cover plate. When we installed the same kind of shields in the full-size assembly, the attachment point was raised to the top of the first afterheater section to prevent direct thermal contact at the cover plate. This change simply involved lengthening of the vertical shield section.

A substantial number of plate growth runs were made to optimize the manipulation of the graphite shields for the subscale setup. These experiments were successfully completed. Only a limited number of experiments, however, were conducted with the modified shield in the full-scale setup. Here, although substantial improvements in plate integrity were noted, it is not clear that the best configuration has been found. We believe that use of these shields is a satisfactory means of optimizing the conditions for growth of crack-free 12-in. plates.

7. Continuous feeder

The initial design of the weldmet rectangular tungsten crucible required a continuous feeding system. Full-scale plate growth required continuous feeding, with material flow rate in the range of 1 to 4 lbs/hr, as shown by Fig. 34 which relates the mass feed rate to the linear growth rate for plates of two thicknesses.

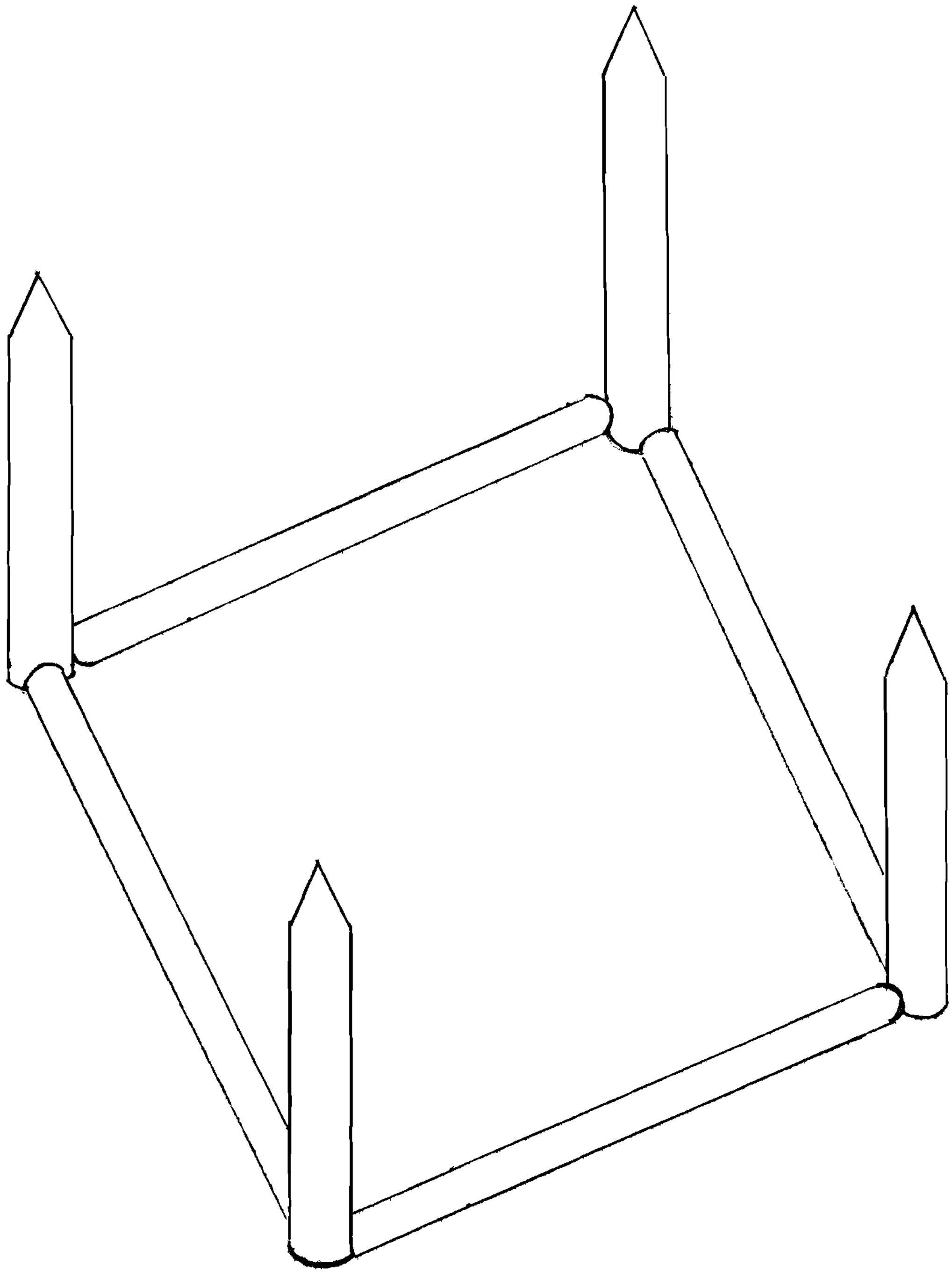


Fig. 31. TIG-welded shield assembly supports

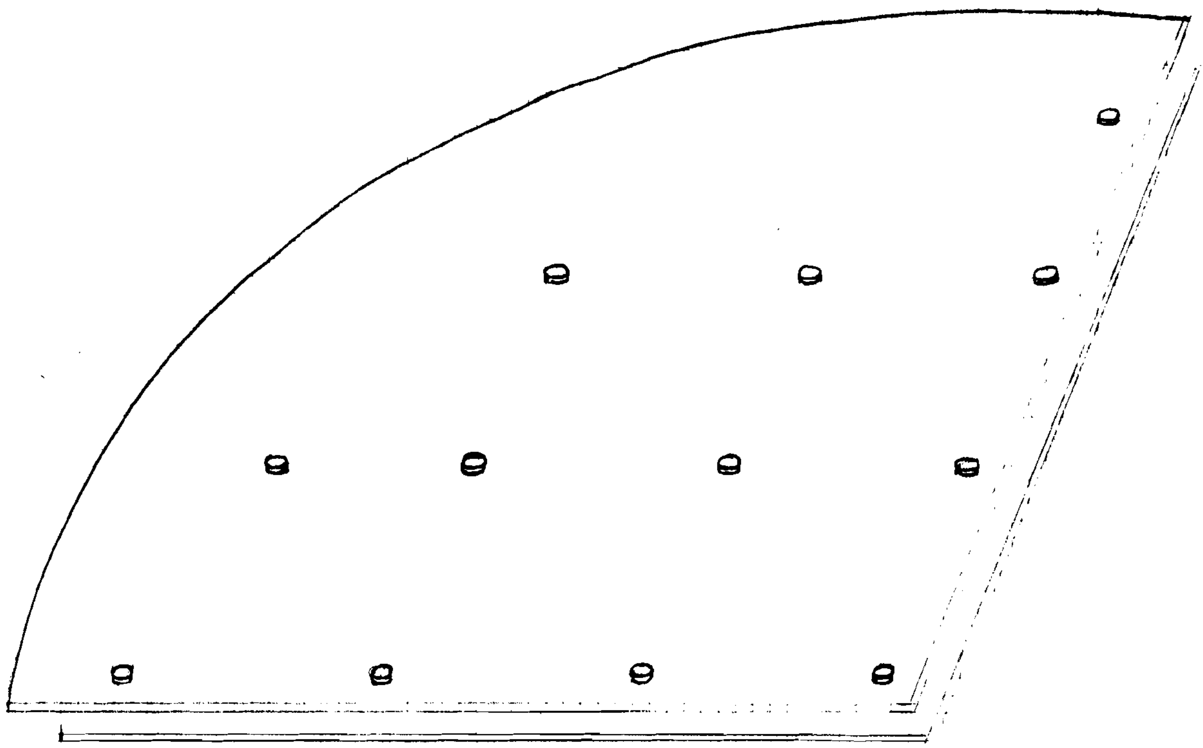
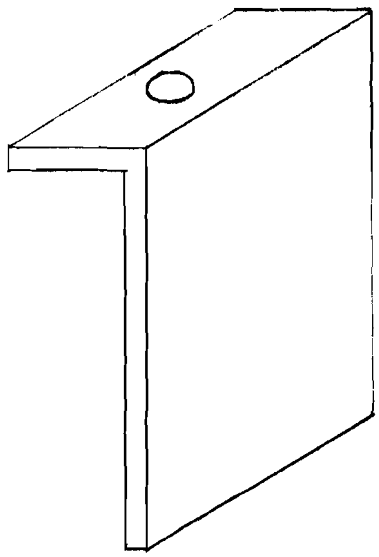


Fig. 32. Tungsten shield and cover assembly



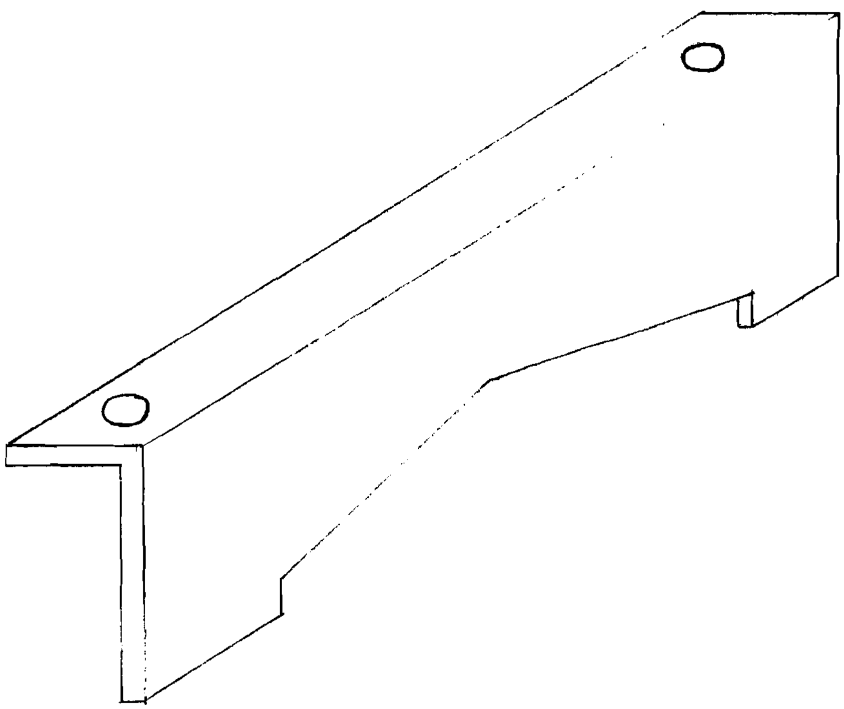


Fig. 33. Grade AUC graphite shields

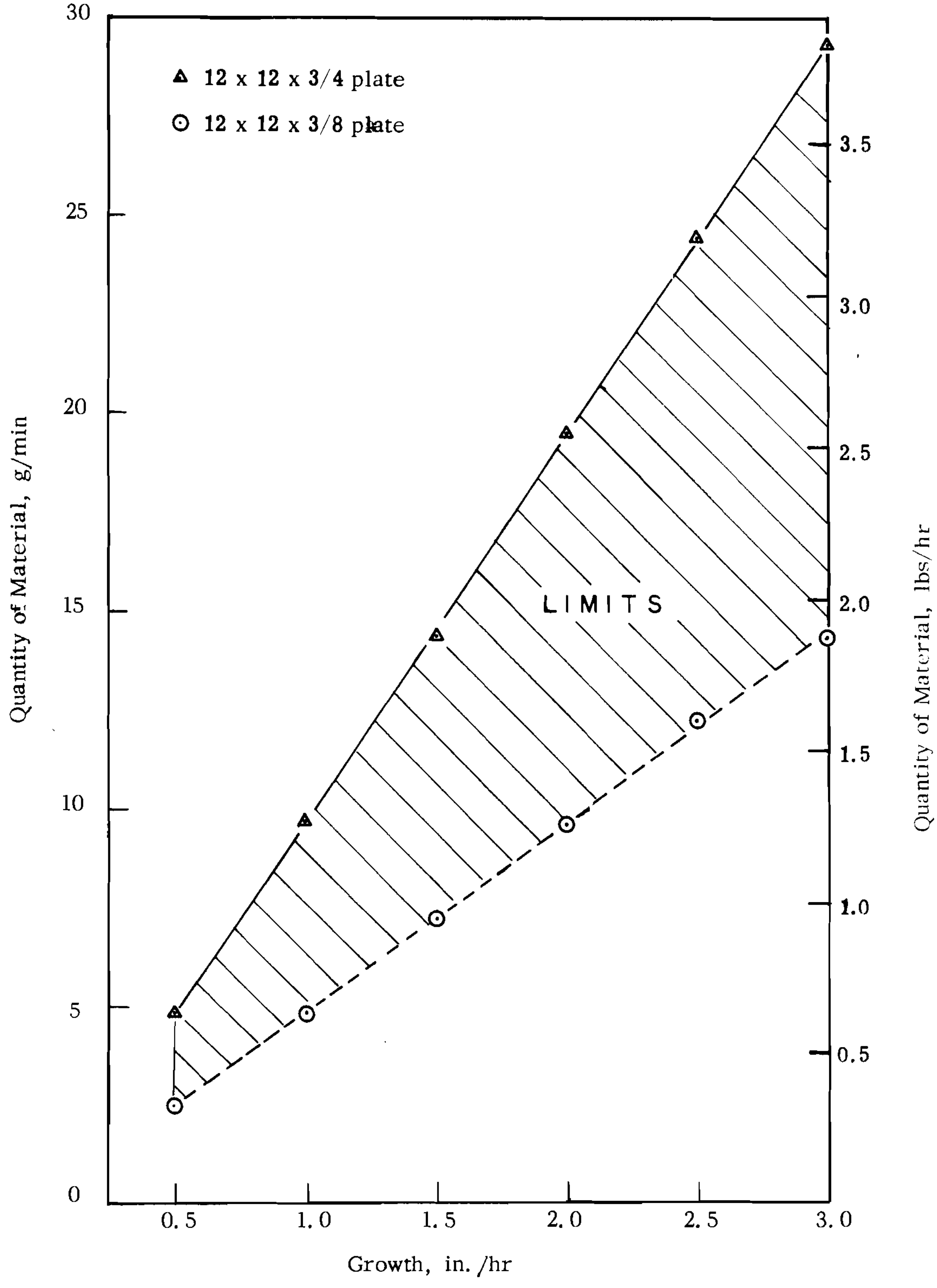


Fig. 34. Mass feed rate for growth of plates

The basic requirements for a feeding system are as follows:

1. Low-cost source material
2. The maintenance of good control to minimize the bulk volume change of molten material
3. Material addition in increments that do not disturb the thermal balance of the system.

The most practical approach for these volume usages per unit time would require the use of the scrap pure sapphire with proper-sized particles. The as-received material is in the form of longitudinally split crystalline sapphire boules. The boules are reduced to granules in the range of 10 to 30 mesh without the addition of impurities. The particles are placed in a hopper and fed to a Syntron feeder, Model EB-00, for continuous feeding of sapphire during plate growth.

The Syntron feeder, Model EB-00, is constructed with a spirally inclined track around its outside perimeter on which the granules are moved by electromagnetically-induced vibration. The drive unit operates at 3600 or 7200 vibrations per minute from 60-cycle alternating current. There are no gears, chains, belts, pulleys, speed reducers, or other rotating, wearing parts that would require frequent maintenance and replacement.

A rectifying element in the control circuit changes the alternating current to a pulsating direct current for energizing the electromagnetic drive. A turn of the rheostat knob increases or decreases the rate of granule movement up and around the spiral track. Due to the coarse adjustment of the rheostat, a powerstat was incorporated into the electrical system to produce fine adjustments and reproducibility of feed-rate settings.

Provisions were made in the design of the furnace enclosure to locate a Syntron, Model EB-000, on each side of the chamber close to the top domed head. However, since use of the larger circular crucibles was adopted, it was not necessary to fabricate and install the designed hopper and Syntron feeder arrangement in the full-scale system.

8. Temperature control

Temperature control to within a few degrees is necessary for optimum growth of large sapphire plates. The temperature control equipment group was built and wired into the high frequency control cabinet. This group contains the necessary transfer circuitry, including a power-operated variable transformer, a proportional-type slow acting controller, a recorder, switches, etc. The controls permit automatic or manual operation, depending on the type of cycle desired for proper sapphire crystal growth. In the automatic control of the temperature, the power is switched between high and low in order to maintain uniform temperature. The Ircon pyrometers are used as the sensing heads.

The temperature control instrumentation was designed and fabricated by Ecco High Frequency Company, North Bergen, New Jersey. A Leeds & Northrup Speedomax H Azar recorder is used in conjunction with a P. A. T. controller mounted in the high frequency control cubicle.

The temperature control unit has one problem which has to be overcome to be a useful tool. At present the sight windows in the chamber tend to cloud after long growth periods. This clouding condition of the windows distorts the true temperature reading and the Ircon sensing head calls for increased power, which results in overheating. The unit has therefore been run manually since the temperature can be stabilized early in the cycle and only minor power changes are required as the sapphire plate increases in size.

9. Annealing system

The results of the tests in the laboratory apparatus indicated that an afterheater section is necessary to prevent the grown sapphire plate from cracking. An additional experiment in the large chamber indicated that here, too, an afterheater was required. The afterheater sections were machined from Airco Speer grade 873 RL purified graphite, designed in sections to make it possible to vary the conditions. The afterheater sections for the full-scale setup are 8 in. long and have a rectangular cross section of 4 in. wide × 17 in. long on the inside with a 1-in. thick wall. Two of these sections, one stacked on top of the other, comprise the afterheater area. The first section, next to the graphite susceptor cover, has a cutout in front for viewing the growing sapphire crystal and orifice and a cutout in the rear for symmetry and a more uniform thermal condition. Three layers of 1/4-in. thick graphite felt insulation are wrapped around each afterheater section. Two thin sheets of graphite 1/8 in. thick are bolted to the top of the afterheater to reduce the open area and heat loss.

10. Pulling mechanism

In the growth of crystals by EFG, as in the growth of crystals from the melt by any other method, the growth speed which can be attained is limited primarily by heat flow. Thus, solidification of an incremental amount of liquid produces heat, from the latent heat of solidification, which must be dissipated. In the growth of large crystals, solidification at the interior of the interface is not quickly dissipated so that the growth rate is appreciably slowed. A range of growth speeds for plate was chosen to be 1 to 4 in./hr. The best plates were pulled at the lower end of this interval ($\sim$1 in./hr). Rates greater than 1.5 in./hr produced unsatisfactory results. Here, however, the problem appeared not necessarily to be dissipation of the latent heat of solidification.

After choosing the range of speeds for the puller, the primary requirements for the puller are for the length of the stroke and for the pulling stroke to be uniform and free from vibration. The pulling mechanism was designed to provide a 24-in. stroke. The mechanism consists of a Beaver ball screw, a Boston Gear spiral miter gear, a Norma-Stoffman bearing, a Bodine reduction motor, a Minarik controller, shafts, bearings, plates, etc. The machining and assembly of parts was accomplished by Tyco. The mechanical puller does not slip, and the combination of the ball screw with the Bodine reduction motor provides a smooth (no vibration) action at any rate of growth. The pulling mechanism shown in Fig. 20, is mounted on the top of the furnace enclosure, sealed with an O-ring clamped between the flanges.

11. Surface polishing

As-grown plates have imperfections in the form of surface waves and require a two-stage polishing operation to produce a smooth surface. This operation consists of first Blanchard grinding, then essentially conventional grinding and polishing. The Blanchard grinding reduces the total cost of the polishing process.

A number of vendors were investigated for the polishing operation. Prices and capabilities varied widely, and we found no vendor to do both operations. Blanchard grinding was carried out by the Syncor Products Co., Malden, Massachusetts., which provided a surface flat and parallel within 0.001 in. Final polishing has been done both at Accumet Engineering Corp., Hudson, Massachusetts, and the Van Keuren Co., in Watertown, Massachusetts. Both provide acceptable quality, but the former is the less expensive of the two.

D. Machine Assembly

The sapphire crystal growing machine was assembled at Acme Industrial Equipment Company, Hingham, Massachusetts, where it was tested for vacuum leaks and repaired by welding under our supervision. The disassembled machine consists of six components: (1) the main furnace enclosure, (2) the main furnace enclosure legs, (3) the vacuum manifold system, including the diffusion pump and valves, (4) the vacuum pump and blower, (5) hydraulic scissors lift, and (6) the electronic control cabinet, placed on skids for transportation to Tyco Labs. After the assembly of the crystal growing machine at Tyco, it was necessary to install the balance of the components which included (1) power feedthrough, (2) high frequency insulated copper work coil, (3) the Tyco crystal puller and controls, (4) the various graphite components and insulation, (5) the power control cubicle, (6) the 175 KW, 960 cycle motor generator set, and (7) the manual reduced voltage starter.

After installation and subsequent tuning of the rf heating equipment, a problem of arcing in the power feedthrough into the furnace chamber arose. It was eventually necessary to pot the two bus bars in the feedthrough using G. E. RTV 30 silicon rubber.

Initial growth trials were conducted using the 34-in. coil, which surrounded and suscepted to both the crucible susceptor and the rectangular chimney-type afterheater. The improved coupling efficiency of the 960-cycle power supply compared to that of the 450-KC supply used in the laboratory equipment, resulted in excessive heat generation within the afterheater. This heat, combined with its proximity to the region immediately above the orifice, resulted in a temperature which exceeded the melting temperature of sapphire, above the orifice.

A second rf coil, 12 in. long, was purchased and positioned in the apparatus such that its top coil was 1-1/2 in. above the top of the crucible susceptor. This coil suscepts only to the crucible susceptor and cover. The afterheater is then heated by conduction from the setup below it. This heating method seems satisfactory. Indeed, the capability still exists to heat the area above the orifice hot enough to melt sapphire and requires careful selection of shielding and placement of components to prevent it.

The original design of a heat barrier below the crucible to prevent excessive heat from reaching the water-cooled pedestal plate had metal shields below the graphite pedestal block that supports the crucible (as shown in Fig. 35) The rods supporting the shields were constructed of tungsten on the top and stainless steel on the bottom. The upper three plates were made of tungsten and had three layers of graphite felt beneath them. Excessive heat reached the stainless steel rods, causing the shield assembly to sag. As a result the water-cooled pedestal plate was overheated and a water leak ensued at the edge.

The heat barrier was subsequently modified twice before a satisfactory arrangement was found. In the first modification, Fig. 35, a set of graded insulators was used, with one component being a porous carbon block. This setup was a satisfactory insulator but produced an unacceptable amount of smoking due to impurities in the porous carbon block.

The final modification is shown in Fig. 19. Six layers of high purity graphite felt, item 16, are placed on the surface of the water cooled pedestal plate. The birdcage support structure, item 3, as shown in Fig. 19, is constructed with grade AUC graphite rings and rods. Grade AUC graphite is a purified grade supplied by Union Carbide Corporation and manufactured for nuclear applications. There are four layers of 1/4 in. thick, high-fired, graphite felt around the outside of the birdcage structure. On the inside of this structure there are five pillars constructed of 1/4 in. thick graphite felt rolled and secured with graphite yarn. These graphite felt pillars support four discs of 1/4 in. thick × 16-in. diameter graphite felt as shown in Fig. 19, item 16. The four 1/4 in. thick felt discs are replaced after approximately three months of continuous operation to insure the proper insulation values required. This last setup is constructed of purified materials and reduces the temperature at the surface of the water-cooled pedestal plate to 75/80°F.

The original solid pedestal support block was also associated with excessive temperatures at the base of the crucible, which reduced the life of the tungsten crucible. Modifications were made to permit the circulation of hot gas through the graphite pedestal support block and around the bottom of the tungsten crucible. The heating of the tungsten crucible is mainly by radiation from the high purity graphite susceptor.

The graphite cover required some modifications to arrive at the desired contour of the opening for proper thermal distribution across the orifice and growing sapphire plate. Graphite felt insulation is used around the susceptor and on the surface of the graphite cover plate. The rectangular afterheater sections rest on the graphite cover to reduce the thermal shock as the growing sapphire plate is raised.

E. Process Optimization

1. Growth apparatus

During the design, construction, and assembly of the large crystal growing furnace, plate growth trials were carried out in the laboratory equipment to establish the design concept for the setup in the big furnace. By means of these trials, we were able to develop the

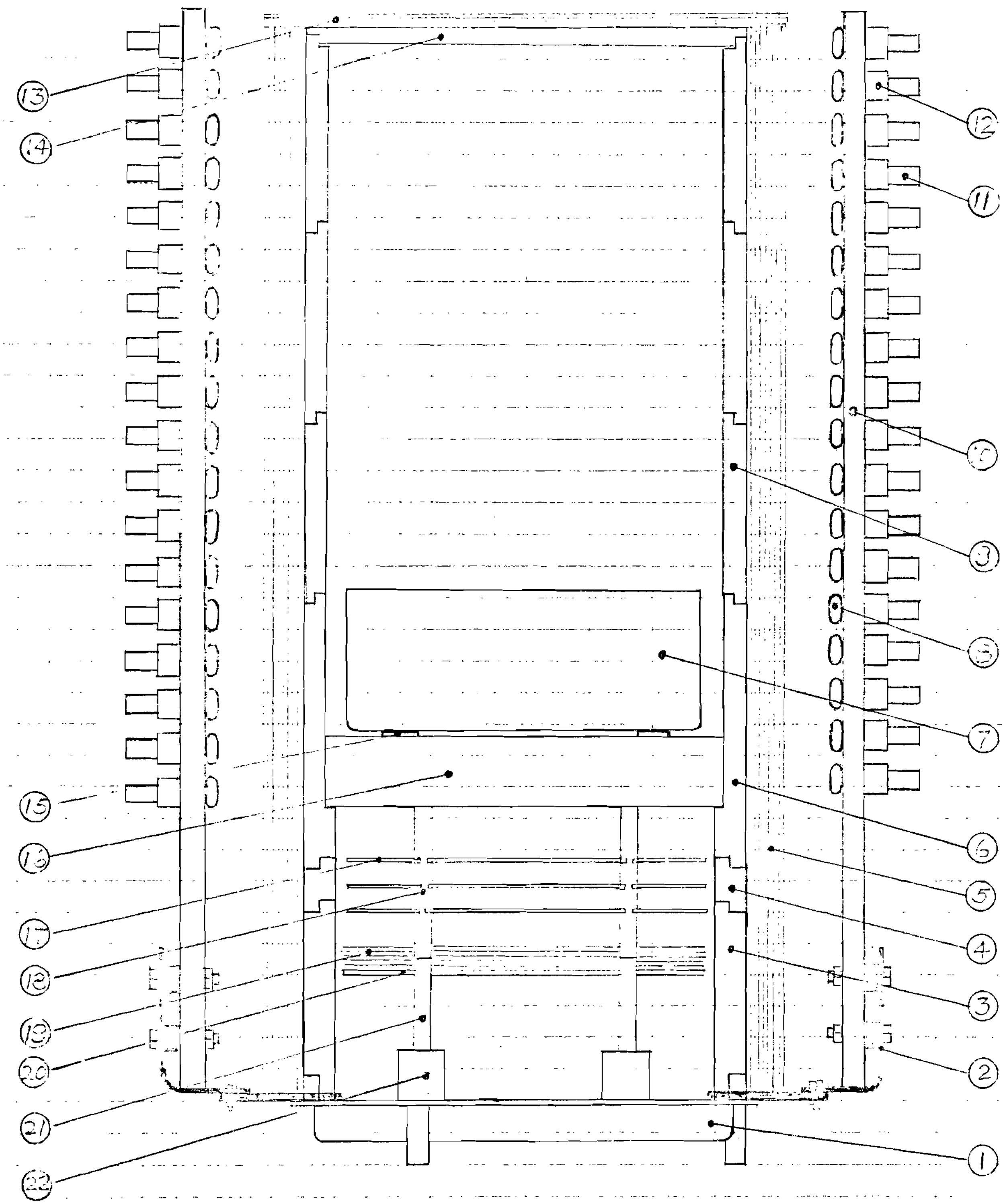


Fig. 35. Sectional diagram for the first susceptor assembly

Fig. 35a. Description

Item No.	
1	Stainless steel watercooled pedestal plate
2	Stainless steel coil mounting brackets
3	Insulating ring-porous carbon 25
4	Coke carbon intermediate ring
5	Graphite felt insulation
6	Graphite susceptor
7	Tungsten crucible
8	Insulated copper work coil
9	Graphite afterheater sections
10	Transite work coil supports
11	Teflon screw caps
12	Transite adjustment blocks
13	Graphite felt insulation
14	Graphite top cover
15	Tungsten crucible shims
16	Graphite pedestal block
17	Three tungsten shield plates
18	Tungsten pedestal rods
19	Graphite felt insulation
20	Stainless steel tie plate
21	Stainless steel pedestal rods
22	Stainless steel pedestal bosses

basis for the susceptor design and for the orifice construction as described previously. In the course of the work on the laboratory equipment, a number of plates up to 3 in. wide were grown and delivered to the Air Force.

The first trials in the assembled large growth furnace served to identify several problem areas. The areas of immediate concern were the support structure beneath the crucible and the temperature distribution above the orifice. The final design of the crucible support structure evolved through two changes. In the first, a porous carbon block was used as both an insulator and support member. Although this arrangement was satisfactory in both of these functions, the setup was found to produce a great deal of smoke. The smoke was eventually traced to impurities, principally iron, in the porous graphite. Subsequently, an open birdcage support of high purity graphite was designed and built. In this structure, graphite felt insulation was used to prevent heat reaching the base of the furnace. The open structure of the support for the crucible allowed circulation of argon beneath the crucible and functioned to preven over-heating of the base of the crucible.

The temperature distribution above the orifice resulted, in the first trials, in producing a temperature above the orifice as hot or hotter than in the crucible. The temperature here was reduced by shortening the coil and by eliminating the afterheater sections. Run No. 52, as shown in Fig. 36, was grown without an afterheater. As the figure shows, the plate could be grown that way but cracked badly as it was withdrawn.

These early trials confirmed various similarities and differences in the requirement for plate growth in the large system as compared to the laboratory apparatus. The main difference between the growth characteristics in the two setups is the altered heating condition, resulting both from the improved coupling between the low frequency motor generator rf set and the various components of the susceptor and after heater, and from the increased amount of heat flow from the much larger heated volume beneath the top of the orifice.

The treatment of these problem areas in the design of the heating system has resulted in a new concept for the total heating system. In the past, the approach to heating the setup for the growth of EFG bodies from the melt has been to use rf coupling to a susceptor at the sides of the liquid bath to supply liquid alumina to the top of the orifice. Heat shields are then placed above the crucible, around the orifice to create the proper gradient for growth just above the orifice. In cases where the resulting gradient caused thermal shock and concomittant cracking as the crystal is withdrawn, an afterheater was added above the crucible to reduce the vertical temperature gradient. This system has proven satisfactory for the growth of small crystals, such as tubing, ribbon, and filament. However, since the pattern of heat flow in this design creates steep horizontal temperature gradients, this type of setup has not proven satisfactory for the growth of large plates, except in the case in which the width of the plate was short compared to the size of the crucible.

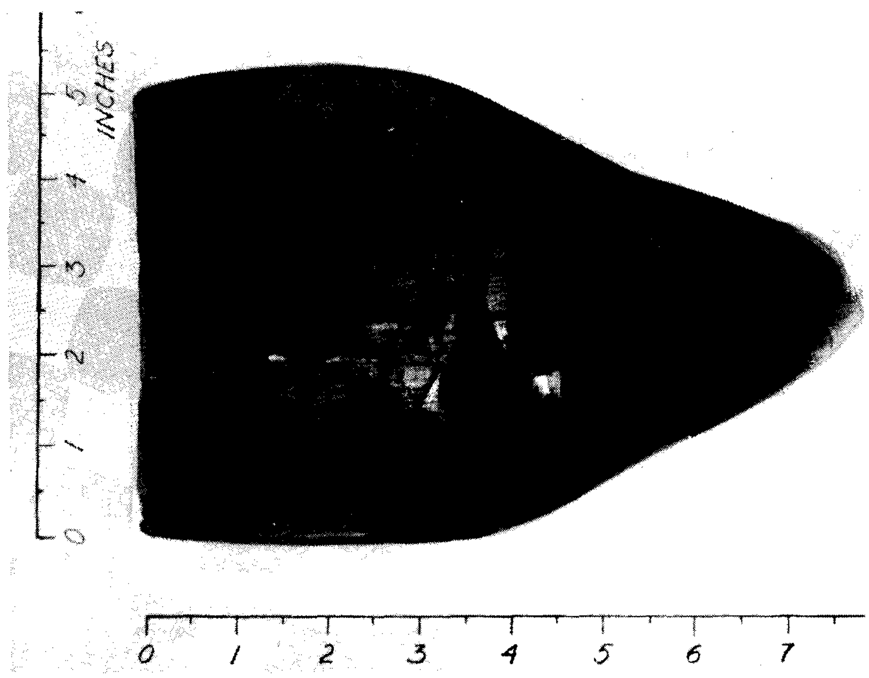


Fig. 36. Sapphire plate, run No. 52, grown without any afterheater

The design concept, as it evolved here, for the heating system is an important design advance for the growth of large linear bodies by the EFG growth method. The essential feature of the new design is that the appropriate temperature and temperature gradients are obtained from a furnace enclosure and modified or controlled by internal heat shielding. In this method, heat loss above and below the setup is minimized to reduce the horizontal gradient. Thus, all of the heat which enters the setup is produced by the rf coupling to the susceptor around the crucible. The afterheater is an integral part of this setup and receives heat by conduction from the graphite cover on top of the susceptor. Graphite felt covers the outside of all parts of the susceptor, cover plate, and afterheater. In addition, graphite insulation under the crucible minimizes downward heat loss from the center of the crucible, while the birdcage structure of the lower support prevents overheating of the lower periphery of the crucible. Heat shielding is used around the orifice, above the crucible, and near the bottom of the afterheater to obtain the proper conditions for growth.

The optimization of the growth process involved first the correction of problem areas such as those described above, and then the optimization of the heat shielding to complete spreading of liquid on the orifice and to maintain the integrity of the plate as it is withdrawn. Optimization of the heat shielding was begun using a subscale orifice and crucible to grow 5.5-in. wide plates which were 0.44-in. thick. This intermediate size was chosen because the problem areas which existed could more effectively be treated first for that size. Work on this size plate was successful and resulted in the development of a process to grow long 5.5- ×0.44-in. plates which were crack-free. Fig. 37 is a photograph of the largest subscale plate which was grown. This plate is 10 in. long.

Growth trials with the subscale orifice and crucible were continued until we had learned enough about the design and use of heat shielding to control cracking. Subsequently, the full-scale setup was assembled for several growth trials. This setup differs in several minor respects from the subscale setup, mainly in the crucible support area. Here, the graphite pedestal block (Item G in Fig. 19) was changed to support the larger crucible. In addition, the 7-in. tungsten crucible had been spaced from the susceptor by means of a graphite ring which was removed for the 15-in. crucible. Finally, the afterheater was also replaced with a larger one for the 12-in. plates.

As anticipated, the first trials with the full-scale setup disclosed that a substantial temperature gradient existed along the orifice. Fig. 38 illustrates the plate grown from the first full-scale setup. This plate was only 10.5 in. wide and had the rippled surface in the center, which is indicative of a cold condition. This experiment demonstrated that further shielding modifications were required since, with the center cold, the orifice extremes were too hot to allow the alumina to freeze. The latter is shown by the failure of the plate to extend the length of the orifice. It should be noted, however, that even under these relatively extreme conditions, the experiment succeeded in producing the largest plate that we had grown up to that time. Further, the plate did not crack during growth, but rather during the shut-down procedure.

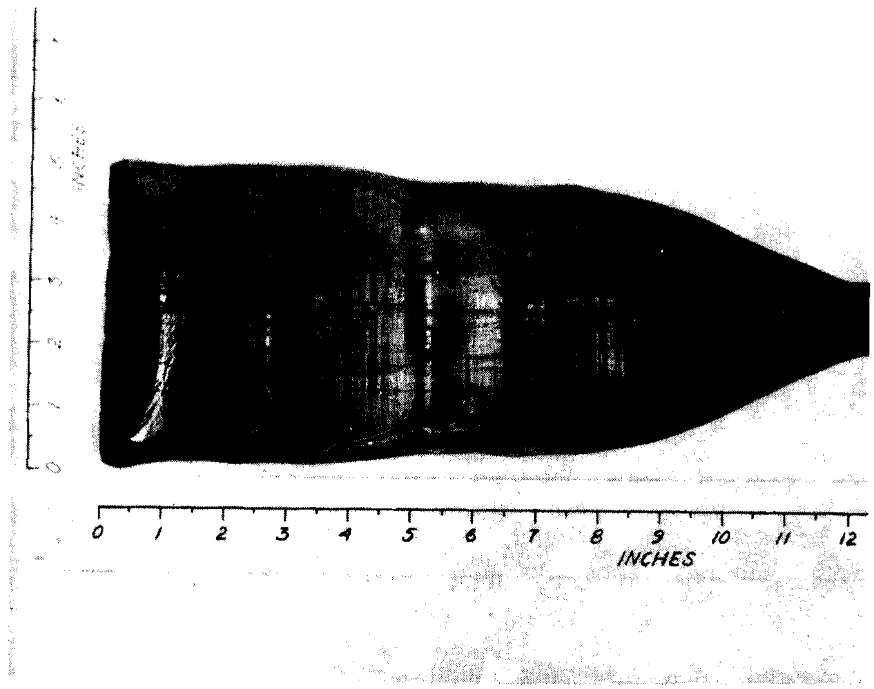


Fig. 37. Sapphire plate, run No. 59 (the growth of this plate represented the successful completion of the optimization of the system for subscale plates)

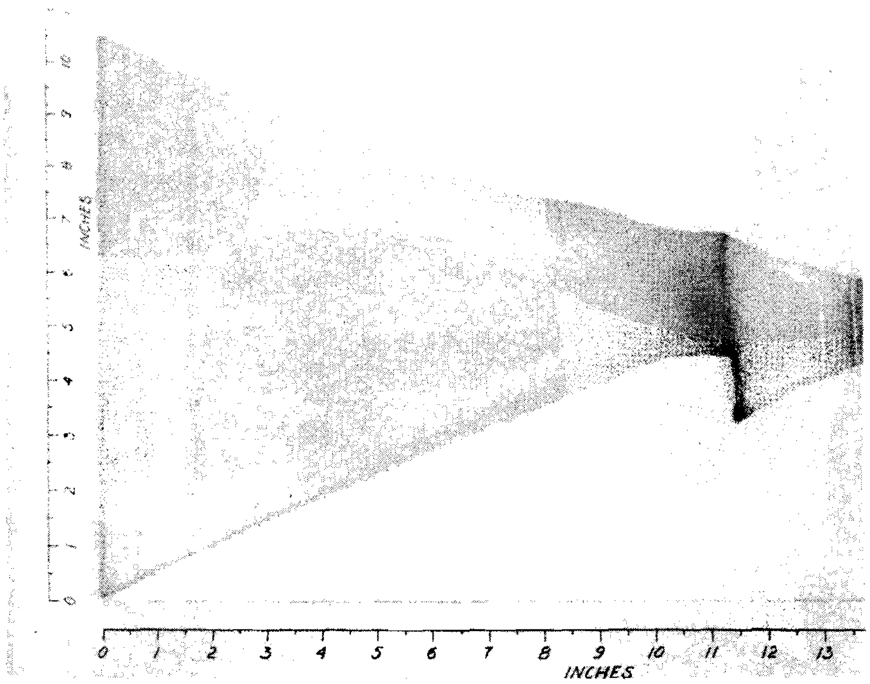


Fig. 38. Initial attempt to grow a full-size sapphire plate

At the end of the program, the shielding was modified in a first attempt to correct the aforementioned difficulties. The subscale setup had employed two types of shields: (1) horizontal tungsten shields beside and near the level of the orifice, and (2) vertical graphite shields which were attached to the graphite cover plate, just below the afterheater. The tungsten shields act to prevent horizontal and oblique radiative heat transfer from the walls toward the orifice.

In the large setup, the latter were necessarily closer to the main source of heat, the susceptor walls. It was felt that a more effective means of shielding would be available by surpending these shields from the top of the first 8-in. afterheater section. In this way, direct conduction of heat into the heat shields via their attachment points could be avoided. This change was carried out and resulted in a marked improvement.

Fig. 30 shows the plate which was grown after modification of the graphite heat shields. This plate grew the full width of the orifice, 12.37 in. Once again, however, a rippled region was noted near the center of the plate which indicates that the center is still not hot enough compared to the extremes. Despite the still-present gradient, however, this plate again grew crack-free and cracked only during cooling.

At the present time, the system for the growth of 5.5-in. wide plates can be considered essentially optimized. The work to optimize the apparatus for the growth of 12-in. plate is not complete. In this respect, we feel that two aspects of the growth require further study. First, the graphite heat shields require additional modification to improve the temperature gradient between the extremes and the center of the orifice. These shields are segmented and can readily be modified to make small changes. Second, the most striking feature of the behavior of the 12-in. plates was the apparent ability of the system to grow crack-free plates, but to somehow cause these plates to crack during shutdown of the furnace. While correcting the temperature across the orifice could be expected to result in plates less likely to crack, it is evident that the shutdown procedure should be investigated to improve the yield.

2. Plate quality

Requirements for plate quality include factors of integrity (no cracking), as discussed above, and also clarity. The plates which we grew from orifices constructed of parallel 0.030- to 0.040-in. molybdenum plates did not satisfy the requirement for clarity, even on a qualitative basis. Other experimental orifice designs produced satisfactory clarity but were more difficult to grow from without cracking. In this section we will discuss the factors which affected clarity, their hypothesized relationship to the orifice design, and how we would propose to eliminate them in the future.

Fig. 39 is a photomicrograph of the structure in plates grown from the standard orifice. The feature of interest here is a series of voids aligned in the growth direction. These stringers of voids form only in that material which solidifies above the apertures between molybdenum plates in the orifice, and they form veils which limit the clarity. In contrast, Fig. 40 shows a plate after polishing which had been grown from an orifice containing only a single, central feed slot. This plate was perfectly clear, both on the macro- and micro-scale.

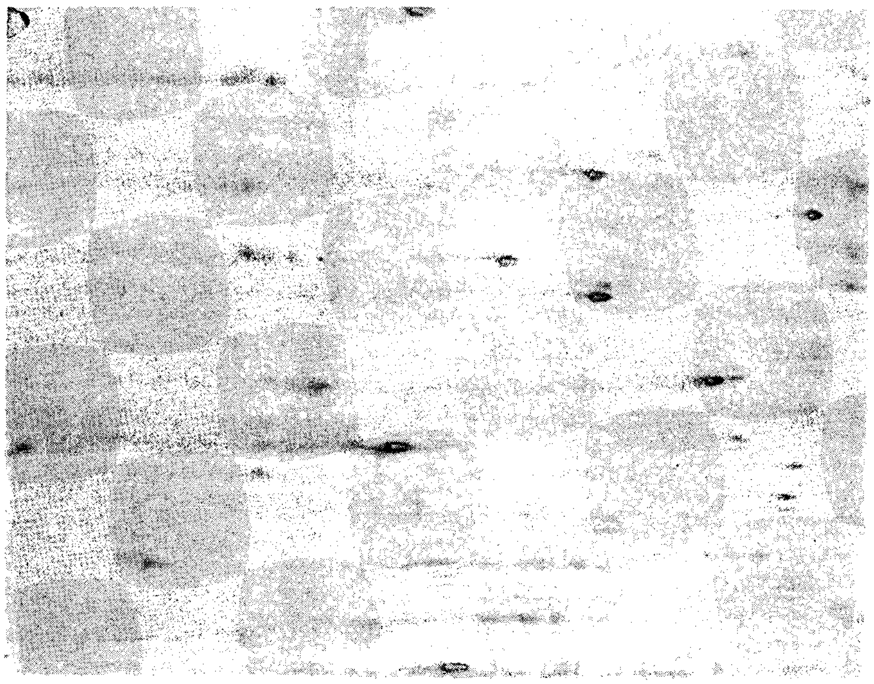


Fig. 39. Photomicrograph of the void structure in a plate grown from the standard orifice (X10)

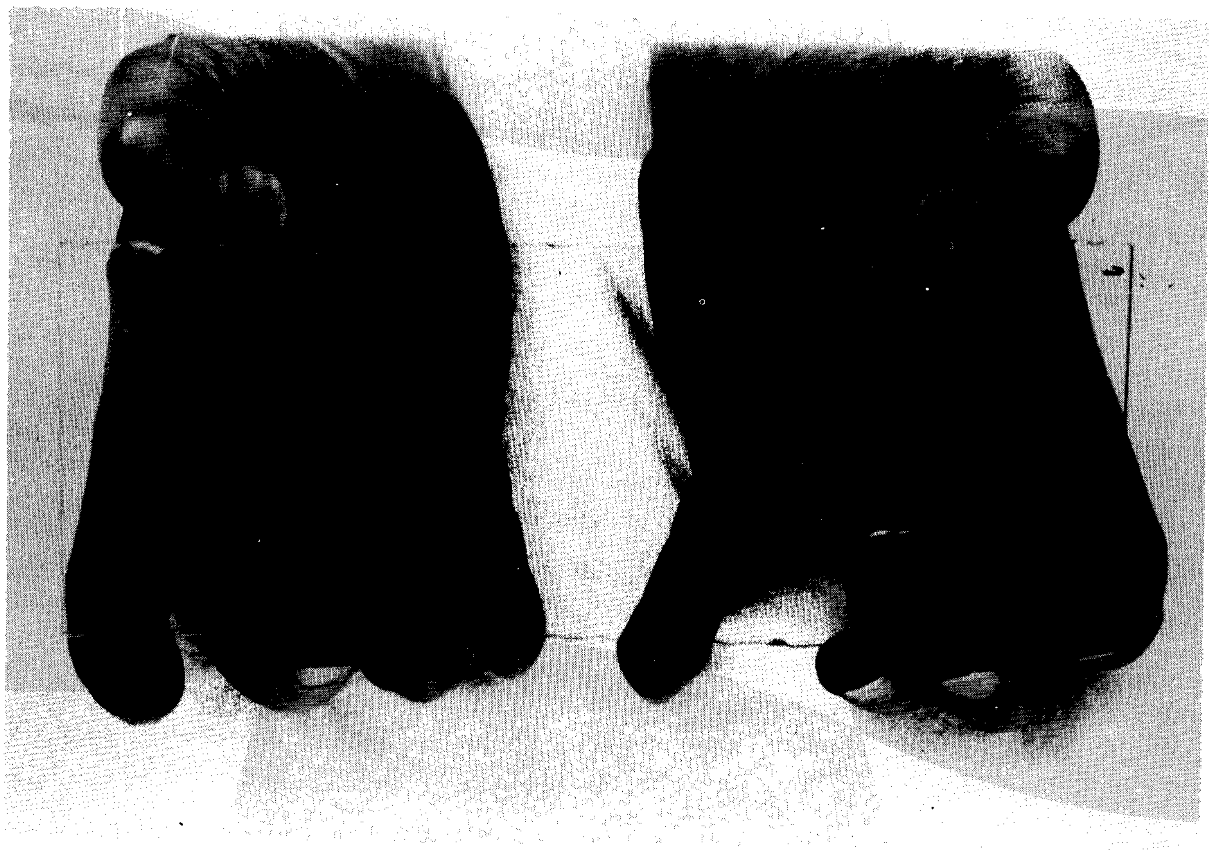


Fig. 40. A clear sapphire plate after rough grinding and polishing (this plate was grown from an experimental orifice containing only a single central feed slot)

In exploring the possible causes for these void veils, we first considered the most obvious causes which are known to produce voids in sapphire crystals. The presence of impurities in the alumina melt can lead to void formation. The mechanism here is related to the thermodynamics of the solidification of impure metals and ceramics. Thus, when an impure material freezes, the composition of the solid in equilibrium with the liquid is different from the liquid, resulting in solute buildup or depletion in the liquid. The solute concentration gradient in the liquid is associated with a gradient in the freezing temperature of the liquid. This gradient is such that the freezing temperature increases along any line normal to the solid-liquid interface, extending into the liquid. Under these conditions it is usual for the freezing temperature of the liquid near the interface to lie above the actual temperature of the liquid. This material is thus constitutionally supercooled, with the result that rapid dendritic or cellular growth into the liquid can occur. When this happens liquid of lower freezing temperature is trapped between dendrites or cells, freezing after the main solifification front has passed. When these areas of entrapped liquid freeze, they shrink, resulting in voids.

Raw material for our growth process is provided by use of scap verneuil sapphire boules. In order to evaluate the possibility that our processing introduced impurities into the pure starting materials, we adopted the use of as-received and cleaned boules, eliminating any crushing or sieving operations normally employed. This change was found not to affect the occurrence of the veils.

Two observations of the solidification behavior support a particular hypothesis for the occurrence of veils of voids. First, the veils always occur over the capillaries. And, second, solidification behavior when the size and shape of the molybdenum plates and feed slots are varied suggests that the liquid transports heat into the meniscus. The hypothesis is that the liquid above the capillaries is hotter than elsewhere, producing upward concavities in the interface in those locations. These concavities would tend to collect gases rejected from the liquid at the interface. Once a bubble had formed, it would move upward as solid grew around it and become a void. Since heat loss upward via the lightpipe effect is an important mechanism for heat loss, the presence of one void should result in higher probability for formation of another one below it. This would result from scattered radiant energy at one void, creating an even deeper concavity at the interface below it.

In an effort to test this hypothesis, and at the same time to examine a means of eliminating the problem, we prepared subscale orifices in which the construction was varied. One method for control of the pore distribution is sharply to reduce the number of capillaries. This method has been successful and was the way by which the plate shown in Fig. 40 was grown. However, orifices of this type have much more severe thermal problems associated with them.

A second, different approach is to taper the upper part of each molybdenum plate to a fine chisel edge while using a larger number of thinner plates. Several attempts to carry out this approach by various techniques did not produce sufficient material for evaluation of the approach.

As a result of these efforts, while we have not positively confirmed the hypothesis, it is nonetheless evident that the void veils which limit clarity can be controlled by means of orifice design. Reducing the feed slots to a single slot at the center of the orifice was the most successful approach that was tried. However, we feel that this is not the only approach to the problem. Clearly, any future efforts to grow plates by the EFG process should begin with a study of the void problem and go on to the thermal problem after the former has been solved.

F. Growth Procedures

This section describes the operation of the equipment for the growth of sapphire plate. Reference is made to appropriate figures in the text for the configuration of various components.

1. Equipment setup

a. The bottom dished head is lowered by releasing the clamps and actuating the scissors lift. After lowering, the head is moved out from beneath the chamber.

b. The seed is attached to the holder. After assembly of the components in the dished head, the head is raised, with an operator, part way into the chamber to set the angular orientation of the seed to be parallel to the orifice.

c. The orifice top is polished to 8 to 16 μ in. after which the metal crucible, liner, heat shield, and orifice components are cleaned.

d. A weighed charge of sapphire is loaded into the liner and the remaining metal components are assembled as shown in Fig. 19. Several sapphire chips are placed on top of the orifice.

e. Heat shielding is fastened to the afterheater, and the cover plate and afterheater are assembled as in Fig. 19.

f. After alignment of the seed, the bottom dished head is raised with the scissors lift and clamped into position. The scissors lift is then lowered.

g. The flexible water and drain lines are attached to the dished head and pedestal base plate. Also, power leads and water connections to the control cubicle are secured.

2. System evacuation and back filling

a. The ballast valve and vacuum line are closed and the mechanical pumps are actuated.

b. The roughing valve is opened and the system is evacuated to 50 microns. Then the roughing valve is closed and the foreline and high vacuum valves are opened.

c. The diffusion pump is actuated by turning on the power to its heaters and starting cooling water flowing through the upper cooling coils.

d. When the indicated chamber pressure is below 10 microns, the high vacuum Penning gauge is actuated.

e. The chamber is pumped to 1×10^{-4} torr after which the high vacuum valve is closed and the power to the diffusion pump turned off. The diffusion pump reservoir is allowed to cool to room temperature and then is isolated by closing the foreline valve.

f. The chamber is now back-filled with argon to not greater than 0.5-Atm pressure.

3. Heating

a. Cooling water to and the main power for the motor generator are turned on. The low voltage starter for the generator is pressed down and held for approximately 50 sec until the unit reaches full speed.

b. Cooling water for the control cubicle, the chamber, and the support pedestal is turned on.

c. The Field and Load switches on the control cubicle are turned on and power to the work coil increased slowly to 175 KW. During this procedure the gauges are checked to make sure that no gauge is more than 10% above the red line. Balancing may be carried out as needed by varying the transformer and capacitor settings.

d. The temperature in the setup is monitored during heatup either by the Ircon indicator or by use of an optical pyrometer sighted through the front viewport. As the temperature approaches the melting temperature, the power setting is reduced.

e. When the sapphire on top of the orifice begins to melt, the power is shut off and the chamber is again evacuated by means of the mechanical pumps. This operation is carried out to remove gas bubbles trapped in the melt.

f. The chamber is again back filled with argon and the power is brought back up to the operating power level.

4. Plate growth

a. The seed is lowered by actuating the puller, and is brought into contact with the top of the orifice. The seed is held here and allowed to equilibriate.

b. The puller is set for the desired growth speed, usually near 1 in./hr. The calibration is shown in Fig. 41.

c. The puller is switched on. The operator observes the seed as it is withdrawn and notes one of the following conditions: (i) seeding is successful. The plate begins to spread towards the ends and edges of the orifice. A thin bright-appearing meniscus is visible at at the junction of the plate and the orifice, (ii) the setup is too cold and the seed freezes to the orifice. No meniscus is visible and the orifice is drawn upward with the orifice, (iii) seeding is

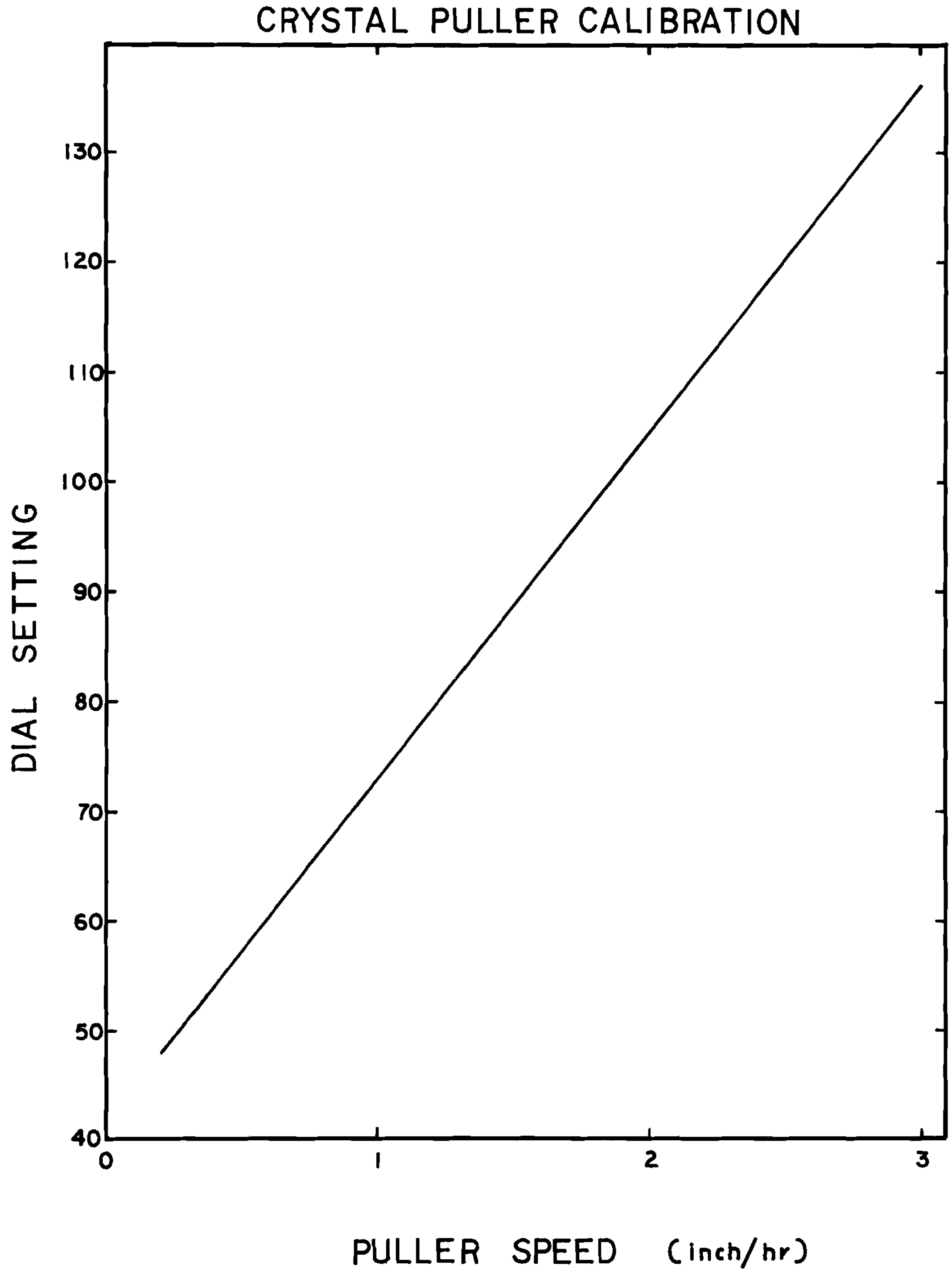


Fig. 41. Crystal puller calibration chart

or is not successful. The setup is too hot and the plate either separates from the orifice or begins to withdraw from the edges.

d. In the event of (i), growth is continued. In the event of (ii), the puller is stopped and the power increased to separate the orifice and seed. In the event of (iii), the power is reduced.

e. During growth, the operator observes the growth at 10- to 15-min intervals, paying particular attention to the presence of a meniscus and any change in dimensions of the growing plate. Power setting is varied accordingly.

f. When growth is completed, the plate is disconnected from the orifice by increasing the speed. Power is gradually reduced over a period of 1 hr.

g. When the power is completely off, the plate is allowed to cool in situ for a period of 6 hr after which the chamber can be opened. Cooling near the end of this cycle can be increased by replacing the argon gas in the chamber with helium. This is done by use of the mechanical pumps, followed by back filling with helium gas.

5. Plate removal

The plate is removed from the setup by following step 1a, and reversing step 1b. The plate is separated from the seed by diamond sawing.

G. Summary

An apparatus for the growth of 12-in. × 12-in. sapphire plates has been designed and assembled. This system represents a basically new approach to attaining the conditions for crystal growth by EFG and is therefore not just a scaleup of the small laboratory apparatus. The new system incorporates a graphite furnace enclosure containing the metal crucible and orifice components used for sapphire plate growth. The furnace enclosure includes a graphite susceptor, graphite cover plate, and graphite chimney-type afterheater which receives heat by conduction through the cover from the susceptor. The temperature distribution in the critical area above the orifice is determined by horizontal tungsten heat shields over the melt and by vertical graphite heat shields near the bottom of the afterheater.

During the optimization of this system, it was found necessary to begin by the growth of subscale 5.5-in. wide × 0.440-in. thick plates in order to determine how shielding could be used to obtain the necessary temperature distribution. After successful solution of these problems, during which several large plates were grown, the technique which had evolved was applied to the growth of 12-in. wide plates. While no new problems arose here, the adaptation to the 12-in. plates was not completed within the contract period. Nevertheless, several cracked 12-in. wide plates were grown and demonstrated steady improvement in quality by minor alterations of the heat shielding.

During the growth of plates of both types, a defect affecting the optical clarity of the plates was observed. The defect has the form of strings of voids which form veils. Experiments were carried out in which the relationship between these veils and the orifice design were investigated. These experiments successfully demonstrated that at least one orifice design modification is possible. By means of this study we successfully grew an extremely transparent plate of substantial size.

At the present time, the design concept for the plate growth system has demonstrated its suitability for the growth of very large sapphire plates. The work which remains here is the development of a new orifice design based on the experimental modification to improve optical clarity, and the further modification to eliminate the remaining cracking in the largest plate size.

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13. ABSTRACT

Progress on concurrent programs to establish manufacturing programs for the growth of single crystal sapphire in two shapes is reported. One process is for the simultaneous growth of 25 high strength continuous sapphire filaments while the other is for the growth of 12 in. × 12 in. transparent sapphire plates. The objective of the first program was to increase the production rate capability and decrease the cost of sapphire filament by designing, building, and operating a multiple filament machine. The successful achievement of these objectives are described, and recommendations for scaleup to further increase production capability and to decrease costs are given. Particular advancements in the technology of several components used in the filament process are reported. The objectives of the second program were to develop a system and to establish a manufacturing process for the growth of large sapphire plates. The design and assembly of a vacuum enclosure and a new concept for heating the growth setup are described. The successful optimization of the equipment and procedures for the growth of intermediate size (5 in. × 10 in.) sapphire plates is discussed, together with the partial achievement of the complete objectives. Analysis of the remaining optimization for the growth of high quality, full-scale plates is reported.

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